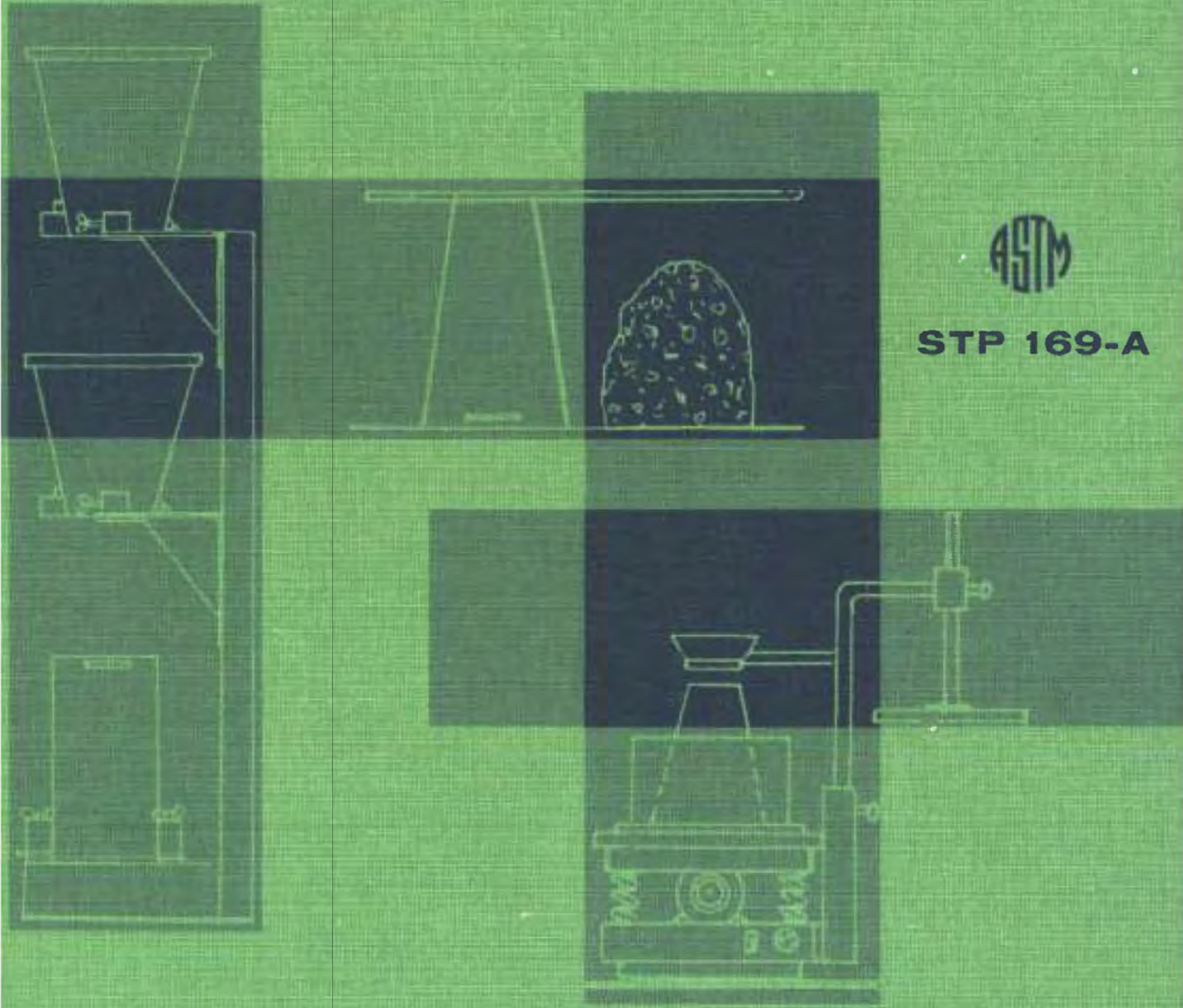
Significance of Tests and Properties of...

CONCRETE
AND
CONCRETE-MAKING
MATERIALS



STP 169-A

AMERICAN SOCIETY FOR TESTING AND MATERIALS

Significance of
TESTS AND PROPERTIES
of
CONCRETE AND CONCRETE-MAKING MATERIALS

An Appraisal of the Properties of Concrete, Concrete Aggregate, and Other Concrete Making Materials (Except Cements), of the Tests by Which They Are Measured, and of Specifications Under Which They Are Selected and Controlled

Reg. U.S. Pat. Off.

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FOREWORD

This publication is a revision and expansion of the report on "Significance of Tests and Properties of Concrete and Concrete Aggregates" published in 1956. That publication in turn replaced editions of "Report on Significance of Tests of Concrete and Concrete Aggregates" published in 1935 and 1943. The present report introduces a still broader scope in that the significance of specifications, as well as of the properties and test methods, is discussed. Moreover, this report covers several types of materials not referred to in the earlier reports but whose importance has increased greatly since preparation of the 1956 report.

As was true for the previous publications in this series, the separate chapters have been prepared by individuals selected because of their knowledge of the respective subjects and because of their participation in development of pertinent methods of testing and specifications. While independent expression by the authors has been encouraged, individual chapters have been reviewed and the entire report has been coordinated by a special committee appointed for that purpose by Committee C-9 on Concrete and Concrete Aggregates. The special committee in charge consists of: R. C. Mielenz, The Master Builders Co., Division of Martin Marietta, Cleveland, Ohio, chairman; D. L. Bloem, Nat. Ready Mixed Concrete Assn. and Nat. Sand and Gravel Assn., Washington, D.C.; L. E. Gregg, L. E. Gregg and Associates, Inc., Lexington, Ky.; C. E. Kesler, University of Illinois, Urbana, Ill.; and W. H. Price, Am. Cement Corp., Los Angeles, Calif.

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RELATED ASTM PUBLICATIONS

Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete, STP 266 (1960), $7.50

Masonry Testing, STP 320 (1962), $6.50

SIGNIFICANCE OF TESTS AND PROPERTIES OF CONCRETE AND CONCRETE-MAKING MATERIALS

INTRODUCTION

Primary functions of the American Society for Testing and Materials are the fostering of research on the effect of physical and chemical properties of materials upon end uses, the establishing of procedures by which significant properties can be evaluated with adequate accuracy and precision, and the fixing of limiting criteria that define the acceptability of materials for specific uses.

Although these objectives are simple in principle, they usually are impossible of exact realization in practice for many reasons. For example, data and theory effecting a general correlation of a property with performance of the material in an end use frequently do not exist. Commonly, appropriate methods of testing have not been developed, or cost of labor or facilities or the required time of testing may render infeasible their application to selection and control of materials. Consequently, most methods of test that are employed for control and evaluation of technical materials are compromises in that they may not measure uniquely with accuracy the properties essential to adequate performance of the material. Rather, the practical tests commonly only generally reflect the qualities of the material, and, moreover, the test may be applicable at all only under a limited range of conditions or to only certain types of materials from among materials that can be considered to be alternatives in the selection for specific uses.

In addition, the specified limits typically are established on the basis of test data that may not be generally applicable or on the basis of experience that does not cover important types or kinds of service or performance. Hence, general specifications may require modification before they can be applied appropriately to specific work.

These facts suggest why technical committees and others concerned with establishing standards controlling selection and use of industrial and engineering materials should periodically set forth the basis of testing and standards of acceptance so as to make clear their value and their limitations. That is, the "significance" of the properties measured, the tests performed, and the criteria stipulated should be described so that the prospective user can decide upon the degree of confidence that is justified with respect to the securing of a desired level of beneficial properties, the avoidance of defects or deleterious qualities, and the applicability of the standards to materials and service conditions of interest to him.

With respect to concrete, Committee C-9 on Concrete and Concrete Aggregates has attempted to meet this need by compilation of three reports,[1] each

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[1] *Report on Significance of Tests of Concrete and Concrete Aggregates, ASTM STP 22*, Am. Soc. Testing Mats., 1935; *Report on Significance of Tests of Concrete and Concrete Aggregates, 2nd edition, ASTM STP 22-A*, Am. Soc. Testing Mats., 1943; *Significance of Tests and Properties of Concrete and Concrete Aggregates, ASTM STP 169*, Am. Soc. Testing Mats., 1956.

comprising coordinated papers that analyze the significance of tests and properties of concrete and concrete aggregates. This report is the fourth of this kind. In the interim since publication of Special Technical Publication No. 169 in 1956, a large amount of new information has become available on the properties of concrete and concrete-making materials, new testing procedures have been developed, many existing procedures have been substantially modified, and new technical areas have been encompassed within the scope of responsibility of Committee C-9.

In 1962 a task group was appointed by the Executive Subcommittee of Committee C-9 to determine whether Special Technical Publication No. 169 should be revised or should be reprinted without change. Based upon a poll of all extant authors of that report and of chairmen of all technical subcommittees of Committee C-9, decision was made to revise the report as required to accommodate new information and concepts and to include chapters covering several properties and materials not treated in the earlier report. Moreover, the scope of the report was increased to include the significance of specifications on concrete and concrete-making materials within the scope of interest of Committee C-9. Development of the new report was carried out under the supervision of a special committee appointed by the Executive Subcommittee.

In most instances, authors of Special Technical Publication No. 169 have prepared equivalent papers for the new report. However, authorship was reassigned for several papers because the author declined the invitation to accept the new assignment, retirement of the author, termination of activity directly involving the subject of the paper, or (in three instances) death of the author. Insofar as possible, the new assignments were given to the chairman or a recent past chairman of the technical subcommittee responsible for research or testing and specifications germane to the section.

The first part of the report comprises five papers dealing with problems and methods of sampling, testing, evaluation of data, and needed research that are important in all phases of selection and control of concrete and concrete-making materials. Because of the importance of plans and procedures of sampling, the first section has been substantially enlarged and recommendations on sampling of diverse concrete-making materials are provided. The paper on Quality of Testing is a new and valuable addition to the report.

Part II of the report deals with tests, properties, and specifications on concrete, as before, but a paper on "The Nature of Concrete" has been included so as to afford clearer insight into the physical and chemical phenomena and internal structural features that determine the properties and response of fresh and hardened concrete.

Freshly mixed concrete is covered by four papers that comprise the scope of five papers in Special Technical Publication No. 169, the subject matter having been consolidated and arranged in a slightly different way.

Seventeen papers give comprehensive coverage of hardened concrete. Theory and evaluation of creep have been included with discussion of elastic properties so as to provide for more effective treatment of testing and research that are within the scope of a single subcommittee. Two new papers cover Corrosion of Reinforcing Steel and Corrosion of Embedded Materials Other Than Reinforcing Steel, these being subjects of considerable current interest and research. Resistance of concrete to fire and phenomena of radiation and shielding are treated in two papers, rather than one, so as to allow opportunity for adequate examination of these fields in which much new information has become available recently.

Under special categories of concrete, a new paper on Packaged, Dry, Combined Materials for Mortars and Concrete has been included to cover the new specifications for and methods of testing of this class of product.

Part III on aggregates is organized in the same manner as was the comparable portion of the former report, but the nine papers have been revised and updated to cover new developments and to incorporate discussion of pertinent specifications.

Part IV comprises six papers dealing with various concrete-making materials other than aggregates. The papers on curing materials, air-entraining admixtures, and mineral admixtures have been revised completely under new authorships, and additional, new papers have been incorporated to cover properties, test procedures, and specifications for chemical admixtures and for organic materials for bonding, patching, and sealing concrete.

Throughout preparation of the papers liaison was maintained among authors dealing with closely related subjects, and drafts of all of the papers were reviewed for accuracy and to avoid overlapping or omission of significant subject matter. Nevertheless, in all instances, the paper is the contribution of the individual author and represents his point of view. Some overlapping is unavoidable and, to some extent, is desirable in order that each paper will be as complete as feasible within itself.

All of the papers were made available for review on request by members of Committee C-9, and the report as a whole was approved by letter ballot of the membership as a publication of the Society under the sponsorship of the Committee.

Special Committee in Charge,
Richard C. Mielenz, *Chairman*
Delmar L. Bloem
Lowell E. Gregg
Clyde E. Kesler
Walter H. Price

PART I

General

TECHNIQUES, PROCEDURES, AND PRACTICES OF SAMPLING OF CONCRETE AND CONCRETE-MAKING MATERIALS

By E. A. Abdun-Nur,[1] Personal Member, ASTM

Millions upon millions of dollars change hands daily in various segments of the concrete industry, based on evidence obtained from samples. Yet, in most instances, the sample is obtained in an indifferent manner by someone who is ignorant of the basic principles of sampling (frequently a laborer or warehouseman), of the use the sample is to be put to, of the testing details to be performed on the sample, and of the final decisions to be based on or derived from the test results—all very important factors that should be taken into account in the sampling process. There appears to be no adequate appreciation of the importance of sampling by those who should be concerned about the problems resulting from poor sampling—those who have to make important decisions and set policy based on test results from samples—this despite the cost of the large volume of samples and testing in the concrete field and the larger economic significance of conclusions which are derived from these.

On the other hand, other industries confronted with similar problems have gone to great pains to develop reliable and efficient methods and procedures. The coal, fertilizer, ore, and abrasives industries [*1–3*][2] which have sampling problems similar to those found, for example, in aggregates, have gone a long ways in developing criteria and methods that increase the probabilities of proper sampling. Basic fundamental principles and approaches that can be of help in developing sampling details for the various segments of the concrete industry have been developed by ASTM Committee E-11 or its members [*4–7*]. Other sampling procedures for concrete and concrete ingredients, that range from indifferent to good, may be found in various ASTM standards (see Appendix) and handbooks and manuals [*8–10*].

What is Sampling?

The Sample:

A sample is a small portion of a larger volume or group of materials, such as a lot, a shipment, a stockpile, a batch, a carload, or truckload about which information is desired. The characteristics of the sample are presented as evidence of the properties of the larger unit from which it is taken. A series of such samples provides a pattern of the variations in properties of the total universe or population. By universe or population is meant the aggregate of lots, stockpiles, carloads, and so on, which comprise any specific material used in a project or produced by a producer under regular production for the prescribed time interval under consideration.

Sampling:

Sampling is the process of obtaining samples from the larger universe or population. Where the universe is perfectly homogeneous, sampling becomes the

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[1] Consulting engineer Denver, Colo.

[2] The italic numbers in brackets refer to the list of references appended to this paper.

simple physical act of lifting a sample from the unit being sampled. Inasmuch as the universe is assumed to be homogeneous, any sample can truly represent the larger homogeneous whole.

Unfortunately, nature in general and the concrete field in particular, rarely if ever, present us with a perfectly uniform and homogeneous universe of any material or process. And, where the rare occasion of perfect homogeneity should occur, one would not know it ahead of time and must, therefore, treat it as a variable. Each lot, truckload, batch, or stockpile, not only varies in some measure from similar units intended to be identically the same, but frequently varies within each such unit. The more heterogeneous the universe, the more work it takes to develop a reliable estimate of its characteristics. Therefore, it pays to save sampling and testing funds where little variability occurs and expend them where more heterogeneity is prevalent.

Sampling is thus much more than the physical act of taking a sample of the larger unit as evidence of the properties of the latter. First of all, a sampling plan must be formulated so as to reflect the variability characteristics of the universe. After that, individual samples taken in accordance with such a plan must be obtained in such a manner that each sample is truly representative of the unit from which it comes.

This broadened concept of sampling is in effect an acceptance of the fact that sampling is a complex business and that the skill needed lies as much or more in the man exercising over-all control of the work as in the man actually taking the sample. This needs a major reorientation of ideas on the subject.

Sampling Plan:

The development of a sampling plan requires the mastery of the sophisticated fundamentals of probability sampling, an intimate knowledge of the product being sampled, and a high degree of skill, experience, background, and creativeness. In some cases, the help of a statistician will prove advantageous in developing the theoretical aspects and procedures for the practical man to follow. Models for probability sampling, significance, and interpretation have been developed by Committee E-11 in ASTM Designations Recommended Practice for Probability Sampling of Materials (E 105), Recommended Practice for Choice of Sample Size to Estimate the Average Quality of a Lot or Process (E 122), and Recommended Practice for Acceptance of Evidence on the Results of Probability Sampling (E 141). The setting up of sampling plans without the use of probability techniques will in most instances introduce subconscious bias.

From the standpoint of economics, sampling plans provide either a cheaper method of achieving a given reliability or result in a better reliability for a given cost. The degree of reliability should be set to fit the economic need or justification. The end result for which samples are taken affects to a large extent the details of the sampling plan.

Once a sampling plan is developed for any given situation or set of conditions, it can be followed easily by the average field man under proper supervision. Details of the many approaches that can be applied to the development of sampling plans for concrete and its various ingredients are beyond the scope of this short discussion. The references listed at the end will provide the reader with a starting point from which to operate.

Taking of Sample:

The actual physical manipulation needed to provide a sample representing a larger unit is simpler than formulating the sampling plan, and the ASTM Books of Standards provide guidelines under the various designations as shown in the Appendix.

The procedures can be mastered relatively easily, and if the instructions are followed carefully, one can expect a reasonably reliable sample to result. Other guidelines are found in various handbooks and manuals [*8–10*]. In general these procedures were developed many years ago, and they are becoming more or less out of step with the new concepts of sampling. They can be improved upon very materially and doubtless will be in the continuing process of reexamination by the various subcommittees.

The relationship of a sampling plan to the actual lifting of a sample may best be illustrated by an example in which the universe is the total concrete of any given class on a project. The concrete reaches the project or is made at the site in a series of batches. Although all batches are intended to be exact duplicates, this is not so in practice. Therefore, for proper evidence of the variations of the concrete properties in the universe there is first needed a plan that designates which of the batches to sample in order to develop the same pattern of variation found in the universe—this is known as random or probability sampling. The second step is to sample each batch which has been tagged for sampling, in conformance with the sampling plan, in such a manner as to reflect the variation pattern in the concrete as it comes out of the mixer. This is best done by taking several subsamples at different stages of discharge from each batch and compositing them for a total sample. In case, on the other hand, it is desired to study the efficiency of mixing of the mixer, then the subsamples are tested separately to develop a variation pattern to determine if such a pattern falls within the tolerances applicable to the situation.

The above example shows strikingly that sampling in the concrete field is not a simple matter and must not be delegated to the untrained or careless, or relegated to a laborer for convenience.

SIGNIFICANCE OF VARIOUS FACETS OF SAMPLING

Economic Significance:

Adequate sampling plans and procedures have more far reaching effects than appear on the surface. In the first place, sampling involves costs of sampling, packaging, and shipping, which represent an investment.

But samples are usually taken for testing which normally costs far more than the sampling and involves time, sophisticated equipment, and skilled personnel, all of which add to the sample cost, and in most instances raise the investment manyfold. However, the greatest economic significance is manifested in the decisions and conclusions based on the test results from the samples—these run into millions of dollars daily in the concrete industry alone. In many cases, such decisions are not only economic factors, but also involve safety and life.

It is thus seen that sampling is the most important link in the whole chain of construction events leading to decisions, exchange of funds between parties, contractual relations, and even safety. Can one, under such circumstances, sufficiently stress the importance of of sampling plans, methods, and procedures?

Random sampling or sampling according to a probability sampling plan permits one to attach measures of confidence to the estimates of population parameters. Biased or subjective sampling precludes the use of such confidence limits. This affects the reliability of decisions based on test results.

Kennedy (p. 90) shows the importance of sampling and of the making of specimens strictly according to the required standards. Waddell (p. 32) stresses the importance of the quality of testing. The question may well be raised whether the quality of testing or the proper making of samples has any significance if the sample has been improperly selected, taken, and handled prior to testing. These are some of the factors that seem to have been frequently neglected if one is to judge by observations of actual sampling in the field.

Too often instructions, written or verbal, require a "representative" sample from a "typical" batch or unit. A sample, for certain purposes as described above, must be representative of the batch or unit, but as far as the universe is concerned, there is no such thing as a single *representative* or *typical* batch or unit; therefore, such instructions are misleading and result in improper samples. Yet, results of tests made on samples taken with such unrealistic instructions are used to make most important decisions even when the data appear most improbable.

Why, then, has sampling been apparently neglected in the concrete field? Many factors have caused this situation to develop.

In the first place in the concrete field, samples are heavy, bulky, and dirty, and therefore, in many cases, the engineer passes the buck by sending someone at the lower echelon to take care of sampling, feeling that he would be wasting his expensive time to do so himself.

Secondly, in the past, when samples were not used to reach conclusions by statistical or probability means, probability sampling was not nearly as important as it is now, when statistics has come into the picture—yet sampling procedures have not been revised.

Thirdly, automation, which makes *product control* possible and places a premium on uniformity has only recently come into being in the concrete field. Uniformity was not a practical aim or achievement as long as most operations were conducted by hand and the influence of the personal differences between individuals was a paramount factor.

Fourthly, decisions have been made to a large extent by whether the engineer felt the job to be satisfactory or not and not so much by test results. The latter were something to stick in the file for legal protection, and when an occasional test turned out to be low, everyone got into a huff and blamed the sample or the contractor and made the latter the goat. No one ever dreamed that occasional low test results are part of everyday patterns of variation and are to be expected. If they fall within the proper variation pattern, they should not be a source of concern. Fortunately these low test results showed up only occasionally as the natural tendency for bias in sampling is towards the better side rather than the poorer side. This problem has been discussed in detail by Abdun-Nur [*11*].

Sampling and Inspection:

In most instances, sampling (the actual taking of the sample) becomes one of the duties of the inspector, which is frequently and unfortunately passed on to a laborer furnished by the contractor. In many instances, it has been observed that the inspector handles sampling as a matter of convenience neglecting the fact that sampling is a most important duty to be carried out in exact accord with specified techniques.

As an example, on one project, written instructions, the specifications, and the formal inspector indoctrination meetings all stressed the obtaining of three sets of cylinders from three distinct batches of concrete to represent the variations in a given volume. Field observations indicated that several inspectors were making the three sets of cylinders, all at one time, from one single batch. This of course yielded erroneous records, inasmuch as the test results were being analyzed as though the tests had come from three distinct batches rather than from one batch. The record thus indicated a higher degree of uniformity than actually existed on the project. Inasmuch as uniformity was tied to strength requirements, this permitted the contractor to get by with lower strengths than desired and assumed for the integrity of the design by the engineer. When an inspector was asked why he was not following instructions, the answer was that "It saves making three messes, and gets the job done all in one mess." In general, when the effect of such disregard of instructions is explained, inspectors are more careful.

Thus, it is not only important that the inspector be adequately trained and qualified to do his job properly and to pay special attention to his sampling plan and procedures, but it is also just as important to have surveilliance to assure that such procedures are carried out properly. Better trained and higher caliber personnel are badly needed as sampling carries a burden of responsibility that has usually been ignored.

Inspection can, of course, be either internal—by the producer or contractor, to control his product—or external—by the owner or his engineer or representative to ascertain compliance with specifications. At the present time, there is need for both to assure proper construction. A detailed discussion of this subject is outside the scope of this paper and has been treated by Abdun-Nur [*12*].

Protection of Samples:

Sampling plans, methods, and procedures are not enough to assure proper samples reaching the laboratory for testing. The proper protection, packaging, and handling of the samples is just as important, as carelessness in these factors can invalidate the usefulness of the best sample. Wills [*13*] has shown the undesirable effects on concrete properties due to the use of bags contaminated with sugar or flour, or bags treated with chemical preservatives.

Every engineer has seen concrete cylinder specimens unprotected, permitted to dry out in the hot sun or to freeze on the job. Occasionally, carelessness has resulted in heavy equipment running over specimens which were damaged beyond usefulness; yet, they were sent to the laboratory for testing with the expectation that usable results would be obtained. Proper protection, prepackaging, and care of samples in the field and in shipping are essential if results are to mean much and if reliable conclusions are to be drawn from such results.

One is amazed to see test data from samples utilized in the making of important decisions when the sampling, care, and protection of samples and testing details are all unknown. This involves serious hazards. Would you want to use data from samples taken in an unknown and probably nonstandard manner? Would you want to base decisions on data obtained from cylinders improperly made, cured, capped, and perhaps tested in a nonstandard manner? Yet data obtained in such a manner are being used every day as a guide for the uninitiated to follow.

Compositing Samples:

The validity of composited samples depends on the purpose and end use to be made of the test results from the samples. If a sample is to be obtained to be representative of the properties of a unit of concrete or aggregate, then subsamples taken from various portions of the unit and composited are in order. But if the sampling is for the study of the variations within a unit, then the subsamples should be kept separate and tested separately. This question of compositing should be decided upon in the development of the sampling plan and passed down to the sampler in the form of instructions to be followed explicitly; otherwise, the analyzed results may be unknowingly very misleading.

Sampling and Product Control:

The control of any product is only possible through proper sampling. This is true whether the product be aggregates, various packaged ingredients that enter into the concrete as constituents at various times, or the concrete itself in the plastic stage. Sampling permits the determination of whether the process is producing what is needed and when adjustments and their extent may be required to bring the process within tolerances and up to the desired quality. To attempt to control a product by occasional sampling of the finished material is unrealistic, ineffective, and too late—by then it has already been produced and cannot in most cases be modified. Yet in the concrete field occasional sampling of this latter type is almost standard routine. It is naive to think that this is product control.

To obtain adequate product control of concrete, every step of the process or procedure must be controlled as well as every ingredient that enters into it. Such control has to be developed by the producer or contractor (who is a producer in the broad sense of the word) and be built into his processes or operations. It cannot be imposed by the purchaser or owner from outside.

The most the owner or purchaser can do is to sample and test to satisfy himself that the product complies with the specification requirements. Incentives built into the specifications by the owner or purchaser are of tremendous help in getting the producer to develop proper product control in his operations.

SAMPLING CONCRETE AND ITS CONSTITUENTS

General:

The Appendix gives a list of the various ASTM designations under the jurisdiction of Committee C-9, or useful in C-9 work, but originating in other committees. The list of references at the end of this paper gives papers, manuals, handbooks, and bibliographies that treat approaches to sampling plans, methods, and procedures in various fields, which can be adapted to the needs of the concrete field. In these references, the reader can also find references to other items in case he desires to go deeper into the subject. Cordon (p. 21) treats of the statistical facets of sampling concrete and its ingredients.

The purpose of sampling will determine to a large extent the desirable sampling plan and procedures. Is the sampling to investigate the quality of a deposit, or a product, or is the purpose to determine the uniformity for a product control operation, or is it to determine whether a material meets specifications requirements?

In general, where an operation is functioning under proper and effective product control procedures, a variable sampling plan can be most effective and economical. In such a plan, a basic full sampling plan and inspection requirement is first developed. When results indicate control and acceptable uniformity for a long period of time, the plan can be reduced to less frequent sampling, but the basic plan is reinstated immediately upon the appearance of any signs that control has been relaxed.

Aggregates:

The work-horse of sampling methods used in aggregate work is ASTM Methods of Sampling Stones, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75) under the jurisdiction of Committee D-4. It treats various procedures for sampling aggregates at various locations such as quarries, pits, railroad cars, bins, and stockpiles. It is quite definite in its instruction and in the cautions about uniformity or variability of the material in the locations being sampled, but not about the tools. It requires the taking of a sufficient number of samples to reflect the variability of the material being sampled but does not indicate how to develop a sampling plan for such a series of samples or under what conditions compositing is valid. ASTM Designations E 105, E 122, and E 141 provide guide lines for developing such sampling plans. Undoubtedly the continuing process of revising Methods D 75 will take some of these ideas into consideration.

The *Handbook for Concrete and Cement* of the Corps of Engineers [*8*], the *ACI Manual of Concrete Inspection* [*9*], and the *Concrete Manual* of the U.S. Bureau of Reclamation [*10*], also contain various methods for sampling, whether natural deposits, finished aggregates, other concrete-making materials, or concrete itself. The references on coal sampling, on bulk sampling, and on sampling in general [*1–7*, *14*, *15*], provide additional information that is applicable, even though not standardized specifically for the aggregate field.

Admixtures, Curing, Patching, Bonding, and Sealing Materials:

These materials reach the job or the concrete products plant in packaged form (cans, buckets, barrels, bags, and so on) or are delivered in some instances in bulk solution form. Others like fly ash (ASTM Methods of Sampling and Testing Fly Ash for Use as an Admixture in Portand Cement Concrete (C 311 – 64 T)) involve the same problems that are found in cement sampling. Obviously the problems of sampling for these products are different than those for aggregates. All of such materials have had some factory processing, and invariably the processor claims very close product control.

In practice, most large users test the source for approval and rely after that on the processor's certification that the material is the same as that submitted for original approval. This is, in many ways, a similar procedure to that used for cement for practically all ordinary jobs except the large government contracts.

The acceptance of such a procedure is based on the false assumption that because it is a processed material there are no significant variations in the final product. This assumption is made for convenience rather than because it represents a true picture—it eliminates the need for continuous costly testing that takes time and can delay the work.

This works fine as long as everything turns out smoothly, but if some problem develops in the concrete, it becomes impossible to determine with any certainty what the significant factors might be. There are not only the normal variations in the materials that are not on record, but also there is always the possibility that some human error somewhere along the line of processing, warehousing, and delivery may have been made and gone undetected. Whether this calculated risk is worth taking is up to the responsible engineer to determine in each case. But if proper original or continuous sampling is desired, the procedures and plans are much simpler than in the case of bulk aggregates.

The proper sampling for these materials becomes a problem similar to that of sampling any manufactured packaged article or item. Many plans have been worked out and proven satisfactory and can usually be adapted to the concrete field [*1–7*, *8*, *10*, *14*, *15*]. Usually this consists of a two-step procedure. The first step is the random sampling of a lot or shipment which results in the selection of a number of the packages out of the whole to be used as evidence of the variation pattern of the lot. The second step is to take individual samples from each package that has been set aside for sampling.

The first step is based on probabilities and statistical principles, while the second step requires manipulative care to make sure that the sample is representative of the container. When the package is relatively small and the product is in the form of a liquid of some type, the second step is easy, as proper shaking or stirring will permit a sample to be taken that represents the whole without much trouble. But when one gets into the larger packages, such as a 50-gal drum, a tank car, or tank truck, then there is always the possibility that there is stratification, and the sample has to be taken in such a manner that it is a composite of the various strata.

Compositing assumes that in the unloading, transferring, and handling the various strata will be intermixed. If this is not the case, then the sampling has to be related to the handling and dispensing procedures in order to develop the variation patterns and is likely to get rather complicated. This can be avoided if the storage container from which the material is dispensed is kept continuously stirred to maintain the uniformity of its contents (ASTM Specification for Liquid Membrane-Forming Compounds for Curing Concrete (C 309)).

In the case of a powdered material that is to be dissolved prior to use, a composite sample from various portions of each of the shipping containers set aside from the lot or shipment for sampling should be taken for the second step of sampling (ASTM Methods of Sampling and Testing Calcium Chloride (D 345) and Specification for Chemical Admixtures for Concrete C 494 – 63 T)). For products such as form insulating mats, curing mats, single use cylinder molds, and so on, procedures have to be adapted for each material. At present, the ASTM Standards contain very little guidance, if any, on the subject (ASTM Specification for Single-Use Molds for Forming 6 by 12-In. Concrete Compression Test Cylinders (C 470 – 61 T)).

Detailed guidelines for sampling these classes of materials, which have proved satisfactory in general industrial applications and which in many cases can be adapted to the concrete field, may be found in the industrial product control literature [*2*, *4*, *7*, *14*, *15*].

Fresh Concrete:

The ASTM standard for sampling fresh concrete is found in ASTM Method of Sampling Fresh Concrete (C 172). This outlines in detail the manipulations in taking a sample from a batch but does not describe sampling plans that permit the selection of the batches to be sampled. This standard is some 10 years old and is all right as far as it goes. It needs revising to bring it in line with present-day thinking and approaches to probability sampling and to provide a broader scope of coverage. In general the same problems that are encountered in bulk aggregate sampling from truckloads are found also in sampling batches of concrete—it is fortunate that the most difficult problem of sampling aggregates from a stockpile does not exist for concrete. Sampling procedures, compositing, details of manipulations, and sampling plans should depend on the purpose for which the sampling is being done.

Hardened Concrete:

ASTM Methods of Obtaining and Testing Drilled Cores and Sawed Beams of Concrete (C 42) governs the taking of cores or the sawing of beams from hardened concrete, but no mention is made of how to decide at what point or points such specimens should be secured. ACI Committee 214 is presently developing a recommended practice which it is hoped will cover this detail, and what is as important, the significance of the tests made on such cores or beams. All this is most important as safety decisions involving potential life hazard, and economic decisions involving large sums are frequently made on the basis of samples from hardened concrete.

A recent example of such problems is a case where a large number of cores was taken and tested by several laboratories indicating a certain level of strength and a variation pattern—all with the exception of one laboratory, where three cores indicated consistently a 50 per cent higher strength than those tested by other laboratories—a most improbable situation if sampling had been properly conducted.

NEEDED IMPROVEMENTS AND THEIR BENEFITS

ASTM Standards:

The existing ASTM Standards contain, in many cases, the manipulative instructions for taking samples of the various concrete ingredients and other materials used in the curing, protection, and repair of concrete, and also for the sampling of hardened concrete. In most cases modifications or revisions and broadening of scope of these procedures are desirable.

Recognition of Variability:

Essentially, the big problem in sampling in the field of concrete technology is the disseminating of the ideas regarding variability. It must become commonly accepted that there is no such thing as *representative batch* or *typical batch*, but rather that a sampling plan that reflects the variations in any given situation, population, or universe is needed. Such a plan provides the proper information for control of concrete and its ingredients, to provide a basis for determining specification conformance, and to permit making realistic decisions when problems arise.

Probability Sampling:

Probability sampling is our biggest need at the present time. To bring it about requires training of many people from the engineer down to the inspector-sampler. The importance of proper sampling and its effect on testing, and on the final most important decisions, needs to be repeatedly stressed. The basic fundamentals for probability sampling are available, and other industries with similar problems, such as the coal and ore industries, have already done a lot of development work adaptable to the concrete field, particularly in the aggregate area [*1–7*].

It has been said that the probability testing of aggregates is of no importance as the testing of the plastic concrete is the final criterion. This may be true for determining compliance with specifications, in the few cases where the latter are based on final performance only. But, for the concrete producer who is trying to control his product, it is most essential that he use the best techniques of probability sampling to determine the pattern of variation of the aggregates or any other ingredients that go into his concrete. This enables him to make the proper allowances in the production of his concrete to meet a given specification requirement.

Inasmuch as the probability approach

to sampling provides the required degree of reliability from the smallest number of samples of any other approach, the reduced quantity of sampling will result in most cases in an over-all decrease of the total cost of sampling. Added to this is the fact that the cost of testing unreliable samples will be eliminated, and the total saving becomes very appreciable.

Of course, the main advantage is that the resulting data are more reliable, and therefore the decisions based on such information become more realistic. This permits designing to closer tolerances or lower factors of safety, and in the long run would result in safer and more economical engineering structures.

Trained Personnel:

Such an approach requires many more sophisticated and properly trained men at various levels, but once plans and procedures have been set, the actual sampling becomes a question of following instructions and being sure that there is adequate supervision of the lower grade personnel who actually lift samples by the more sophisticatedly trained personnel. Thus, the cost of supervision and higher grade personnel might increase, but this will be compensated for by the lower cost of the less skilled samplers.

SUMMARY

Sampling is probably the most important step in the testing sequence on which information, millions of dollars, change hands daily—yet it is the most neglected activity in this area of engineering.

The one thing needed above all else is the realization that variations in composition and properties of concrete and concrete making materials are basic and can only be reduced but never completely eliminated. The degree of uniformity that can be attained in practice depends on the cost of reducing variability as compared with the benefits derived therefrom.

Probability sampling and sampling plans are essential to the determination of the true pattern of the existing variations, and fundamental to the problem of controlling any of the products going into concrete and of the concrete itself.

Control must be made an integral part of every step in production, and cannot be imposed from outside by the job owner or the purchaser of over-the-counter products. All the owner or purchaser can do is to determine whether the material as produced does or does not meet specifications. Incentives built into the specifications can motivate the producer to develop proper control.

It is necessary to sample not only at all steps of production to guide production procedures, but sampling should also be done on the finished product as close as possible to the point of use to ascertain compliance with specifications. Handling of the materials after they have been sampled needs much attention, otherwise the samples will not reflect the materials as they are incorporated in the structure.

More detailed instructions and standards can provide better guidance. Better qualified and trained personnel, under proper supervision, are a necessity to achieve maximum reliability with minimum sampling as a basis for final important economic and safety decisions. This would improve the present sampling situation immensely.

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APPENDIX

ASTM DESIGNATIONS UNDER JURISDICTION OR RELATED TO WORK OF COMMITTEE C-9

ASTM Designation	Title	Sample Origin or Requirement[a]
	SAMPLING AND TEST METHODS	
C 29 – 60	Unit Weight of Aggregate	D 75
C 30 – 37	Voids in Aggregate for Concrete	D 75
C 31 – 62 T	Making and Curing Concrete Compression and Flexure Test Specimens in the Field	C 172
C 39 – 61	Compressive Strength of Molded Concrete Cylinders	C 31 or C 192
C 40 – 60	Organic Impurities in Sands for Concrete	D 75
C 42 – 62	Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths	yes
C 70 – 47	Surface Moisture in Fine Aggregate	D 75
C 78 – 59	Flexural Strength of Concrete (Third Point Loading)	C 31 or, C 192
C 85 – 54	Cement Content of Hardened Portland Cement Concrete	yes
C 87 – 63 T	Effect of Organic Impurities in Fine Aggregate on Strength of Mortar	D 75
C 88 – 63	Soundness of Aggregates by Use of Sodium or Magnesium Sulfate	D 75
C 116 – 60 T	Compressive Strength of Concrete Using Portions of Beams Broken in Flexure	C 78 or, C 293
C 117 – 62 T	Materials Finer Than No. 200 Sieve in Mineral Aggregates by Washing	D 75
C 123 – 63 T	Lightweight Pieces in Aggregate	D 75
C 124 – 39	Flow of Portland Cement Concrete by Use of the Flow Table	C 172
C 127 – 59	Specific Gravity and Absorption of Coarse Aggregate	D 75
C 128 – 59	Specific Gravity and Absorption of Fine Aggregate	D 75
C 131 – 55	Abrasion of Coarse Aggregate by Use of the Los Angeles Abrasion Test	D 75
C 136 – 63	Sieve or Screen Analysis of Fine and Coarse Aggregates	D 75
C 138 – 63	Weight Per Cubic Foot, Yield and Air Content (Gravimetric) of Concrete	C 172
C 142 – 55 T	Clay Lumps in Natural Aggregates	D 75
C 143 – 58	Slump of Portland Cement Concrete	C 172
C 156 – 55 T	Water Retention Efficiency of Liquid Membrane-Forming Compounds and Impermeable Sheet Materials for Curing Concrete	no
C 157 – 60 T	Volume Change of Cement Mortar and Concrete	yes
C 172 – 54	Sampling of Fresh Concrete	yes
C 173 – 58	Air Content of Freshly Mixed Concrete by the Volumetric Method	C 172
C 174 – 49	Measuring Length of Drilled Concrete Cores	C 42
C 192 – 62 T	Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory	D 75 yes
C 215 – 60	Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens	C 192
C 227 – 63 T	Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar Bar Method)	D 75 yes
C 231 – 62	Air Content of Freshly Mixed Concrete by the Pressure Method	C 172
C 232 – 58	Bleeding of Concrete	C 172 C 192

ASTM Designation	Title	Sample Origin or Requirement[a]
C 233 – 63 T	Air Entraining Admixtures for Concrete	D 75 C 192 no
C 234 – 62	Comparing Concretes on the Basis of Bond Developed with Reinforcing Steel	C 192 no
C 235 – 62 T	Scratch Hardness of Coarse Aggregate Particles	D 75
C 289 – 63 T	Potential Reactivity of Aggregates (Chemical Method)	D 75
C 290 – 63 T	Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water	C 192
C 291 – 61 T	Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water	C 192
C 292 – 63 T	Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine	C 192
C 293 – 59	Flexural Strength of Concrete (Center Point Loading)	C 31 C 192
C 310 – 61 T	Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water	C 192
C 311 – 61 T	Sampling and Testing Fly Ash for Use as an Admixture in Portland Cement Concrete	yes
C 342 – 61 T	Potential Volume Change of Cement-Aggregate Combinations	D 75
C 360 – 63	Ball Penetration in Fresh Portland Cement Concrete	C 172
C 403 – 63 T	Time of Setting of Concrete Mixtures by Penetration Resistance	C 172 C 192
C 418 – 58 T	Abrasion Resistance of Concrete	no
C 441 – 63 T	Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to the Alkali-Aggregate Reaction	yes
C 469 – 63	Static Young's Modulus of Elasticity and Poisson's Ratio in Compression of Cylindrical Concrete Specimens	C 31 C 192
C 495 – 62 T	Compressive Strength of Lightweight Insulating Concrete	C 172
C 496 – 62 T	Splitting Tensile Strength of Molded Concrete Cylinders	C 31 C 192
D 75 – 59	Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials	yes
D 345 – 48	Sampling and Testing Calcium Chloride	yes
E 97 – 55	Directional Reflectance of Opaque Specimens by Filter Photometry	no
	SPECIFICATIONS	
C 33 – 63	Concrete Aggregates	D 75
C 94 – 63	Ready Mixed Concrete	C 172
C 171 – 63	Waterproof Paper for Curing Concrete	no
C 260 – 63	Air-Entraining Admixtures for Concrete	no
C 309 – 58	Liquid Membrane-Forming Compounds for Curing Concrete	yes
C 330 – 60 T	Lightweight Aggregates for Structural Concrete	D 75
C 331 – 59 T	Lightweight Aggregates for Concrete Masonry Units	D 75
C 332 – 61	Lightweight Aggregates for Insulating Concrete	D 75
C 350 – 63 T	Fly Ash for Use as an Admixture in Portland Cement Concrete	C 311
C 387 – 60	Packaged, Dry, Combined Materials for Mortar and Concrete	C 192
C 402 – 63 T	Raw or Calcined Natural Pozzolans for Use as Admixtures in Portland Cement Concrete	C 311
C 440 – 61	Cotton Mats for Curing Concrete	no
C 470 – 61 T	Single-Use Molds for Forming 6 by 12-In. Concrete Compression Test Cylinders	yes
C 494 – 63 T	Chemical Admixtures for Concrete	yes
D 98 – 59	Calcium Chloride	D 345

ASTM Designation	Title	Sample Origin or Requirement[a]
	RECOMMENDED PRACTICES	
C 295 – 54	Petrographic Examinations of Aggregates for Concrete	D 75 yes
C 457 – 60 T	Microscopical Determination of Air-Void Content, Specific Surface, and Spacing Factor of the Air-Void System in Hardened Concrete	yes
E 105 – 58	Probability Sampling of Materials	ina
E 122 – 58	Choice of Sample Size to Estimate the Average Quality of a Lot or Process	ina
E 141 – 61	Acceptance of Evidence Based on the Results of Probability Sampling	ina

[a] NOTES:

1. This column indicates the best available source or method of sampling for the particular designation available in existing ASTM standards.
2. Where "yes" appears in the column, it indicates sampling instructions in the specific test method itself.
3. Where "no" appears in the column, no ASTM designation for sampling the material in question is available.
4. Where more than one reference is shown in the column, each such reference reflects one of the ingredients needed for the test method.
5. Where specific sampling procedures are inapplicable, this is shown by "ina" in the column.

SIZE AND NUMBER OF SAMPLES AND STATISTICAL CONSIDERATIONS IN SAMPLING

By W. A. Cordon,[1] Personal Member, ASTM

Variations in samples of concrete and concrete making materials make it difficult to obtain samples with the assurance that they are completely representative of the source or production of these materials. Test results are more reliable as the number and size of samples increase, but additional samples or samples of greater size increase the cost of testing. It is necessary, therefore, to establish the accuracy desired in each case commensurate with funds and facilities available.

It may be better to make no tests than to make tests with poor samples which do not portray the actual properties of materials. An engineer or architect who must rely on samples or tests that do not accurately represent materials or structures could probably make more objective decisions if there were no samples or tests available. He could at least make conservative assumptions rather than rely on fallacious information.

For convenience in discussing statistical considerations, tests for concrete and concrete materials are separated into one or more of the following general categories:

1. *Acceptance Tests*—Tests which demonstrate that the material in question will meet specific requirements of the work as specified. Examples: Grading of aggregate, soundness of aggregate, slump, and air content of concrete.

Performance tests may also be used as acceptance tests. These tests demonstrate how a given material will perform in concrete under field or simulated conditions. Examples: Freezing and thawing of concrete prisms, expansion of concrete prisms due to alkali reactivity, strength of concrete, drying shrinkage, and rate of hardening.

2. *Construction Control Tests*—Construction control tests are made at intervals throughout construction of a project. These tests not only provide a check on the performance of materials and may be used for acceptance or rejection of the work, but they also measure the uniformity of concrete produced. The strength of 6 by 12 cylinders is universally accepted as a standard of control in the United States, since most properties of concrete and concrete materials, as well as the influence of testing and construction practices, are reflected in the strength of concrete cylinders.

3. *Research Procedures*—Tests which are conducted in the laboratory or field to establish relationships among the variables which influence the properties of concrete. Examples: Relationship between strength and water-cement ratio, influence of admixtures on the properties of concrete. Rate of hardening at various temperatures.

The number of samples required and statistical considerations are different for concrete and each concrete material and will be discussed separately.

Concrete Aggregates

Acceptance tests for concrete ag-

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gregates establish the suitability of a deposit or source or quantity of finished production. During preliminary reconnaissance of available deposits, aggregate quality evaluation may be based on test results of a composite sample. This is possible since the general characteristics of aggregate quality can be estimated from a minimum of tests. After preliminary analysis, the properties and variations in the most promising source can be established with samples from locations throughout the area and depth of the deposit.

As a general rule, aggregate samples need only be large enough to include a representative portion of all materials and to provide ample material for all tests contemplated as specified by standard test procedures. The number of samples will vary with the size of the deposit, and the size of samples will vary with maximum size of aggregate occurring in the deposit. For gradation analysis, for example, samples should be large enough to assure occurrence of particles of the largest dimension in sufficient number so that the inclusion or exclusion of one of these large particles will not affect the grading. Variations in the size of samples required for petrographic examination are shown in Table 1 [*1*].[2]

TABLE 1—SIZE OF SAMPLE REQUIRED FOR PETROGRAPHIC EXAMINATION.

Size Fraction	Weight of 300 Particles
3 to 1½ in.	57 lb
1½ to ¾ in.	19 lb
¾ to ⅜ in.	2.6 lb
⅜ to $\frac{3}{16}$ in.	0.75 lb
No. 4 to 8	15 g
No 8 to 16	2.1 g
No. 16 to 30	0.28 g
No. 30 to 50	0.033 g
No. 50 to 100	0.0066 g

Performance tests are gaining favor in determining the suitability of concrete aggregates. These tests are based on comparative performance of concrete aggregates in standard concrete or mortar specimens. This type of test is particularly valuable in determining the influence of aggregates on the resistance of concrete to freezing-and-thawing action and the disruptive expansion caused by the reaction between certain aggregates and the alkalies in cements. These tests require much larger samples, containing as much as 600 lb of graded aggregates. It is not necessary to make such tests with all samples, however. One set of tests with a composite sample is sufficient to indicate these characteristics for each source of aggregate.

Acceptance of an aggregate source does not automatically approve all aggregates from the source, and check tests are required periodically as the aggregates are received for compliance with specification requirements. The number of tests required to enable one to control the quality and uniformity of concrete aggregates during construction will depend to a large extent on the variations encountered. Aggregates from a given source are comparatively uniform with respect to specific gravity, absorption, soundness, abrasion resistance, and their influence on durability and alkali reactivity. Representative samples are tested for these properties at the beginning of the job. Aggregate grading, the quantity of silt and inferior material which can be controlled by processing, may vary as each different section of a deposit is excavated. Daily checks of these properties may be desirable, and where the variation in sand grading is excessive or is controlled by classifiers, continual checks may be necessary. Each shipment of aggregate from a commercial source will require check tests unless quality and uniformity

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

have been established as the aggregate is produced. Many producers control aggregate grading and unsound and deleterious materials during production.

Free moisture in concrete aggregates, particularly the sand, is a major source of variation in concrete strength. Unless the amount of moisture in the aggregate is uniform, frequent tests for moisture content are essential for uniform operation of a mixing plant. Success has been experienced with equipment that continually indicates the moisture content of sand in a batching plant. This equipment is valuable in adjusting the water content of concrete mixes, although frequent calibration tests by standard methods may be required.

OTHER MATERIALS

Materials used in making concrete other than portland cement and concrete aggregates include water and admixtures. Other materials, used to treat hardened concrete surfaces, are also discussed.

Water:

It is generally taken for granted that available water will be suitable for making concrete. Water may contain harmful salts, however, and where there is a question, a chemical analysis of a water sample is made to establish its suitability.

Under this procedure it is assumed that the chemical composition of a source of water will not vary during the construction of the project.

Mineral Admixtures:

Pozzolans and other mineral admixtures may be proprietary materials or may come from natural deposits.

A natural deposit will require sufficient samples to establish the extent and depth of the deposit. ASTM Methods of Sampling and Testing Fly Ash (also used for natural pozzolans) for Use as an Admixture in Portland Cement Concrete (C 311 – 64 T) requires an 8-lb sample for each 100 tons of material.

Chemical tests, physical tests, and performance tests for compressive strength and drying shrinkage are required.

Statistical considerations in testing mineral admixtures requires establishing a reliable average for the deposit, lot, or shipment under test and the uniformity from sample to sample.

Chemical Admixtures:

Nearly all chemical admixtures are proprietary materials sold under a given brand name. With minor exceptions the suitability of chemical admixtures is established by their performance when mixed with concrete. ASTM Specification for Air-Entraining Admixtures for Concrete (C 260 – 65 T) and Specification for Chemical Admixtures for Concrete (C 494 – 65 T) give specific instructions for size and number of samples to be taken.

Statistical considerations are concerned with the sample being representative of the lot or production to be used on the specific work.

Curing Materials:

Membrane curing compounds are used to retain the mixing water in concrete until curing has taken place. These materials are composed of various combinations of chemicals and are sold under various trade names. The performance of these materials is the responsibility of the manufacturer. ASTM Test for Water Retention Efficiency of Liquid Membrane-Forming Compounds and Impermeable Sheet Materials for Curing Concrete (C 156) determines the performance of curing materials in preventing evaporation. This test establishes the acceptance of the material and is assumed to be representative of the production or lot under consideration. ASTM Specification for Liquid Membrane Forming Compounds for Curing Concrete (C 309) specifies samples be taken for each lot, batch, or other unit of production and shall represent a maximum of 2000 gal.

Bonding and Patching Materials:

The use of bonding and patching materials, particularly epoxy resins, has increased in importance in the past several years. The composition of these materials is complex and is varied to produce specific desired results. It is necessary to rely upon the manufacturers recommendations when using these materials.

Sealing Materials:

The application of sealing materials to hardened concrete reduces the absorption of moisture and is a promising method of reducing freezing and thawing deterioration. Proprietary materials for this purpose are on the market and in addition, combinations of 50 per cent linseed oil and 50 per cent thinner are recommended in the literature. There are no standard tests devised to measure the effectiveness of sealing materials in reducing absorption.

CONCRETE

Acceptance and Performance Tests:

Acceptance and performance of concrete is established by tests of fresh concrete and hardened concrete. Tests of fresh concrete provide a means of checking the proportions and properties of the concrete mixture. The slump test and other measures of consistency indicate the water content and, indirectly, water-cement ratio of a given mixture and may be used to reject concretes having excessive water. The measurement of the air content (ASTM Recommended Practice for Microscopical Determinations of Air-Void Content, Specific Surface, and Spacing Factor of the Air-Void System in Hardened Concrete (C 457 – 60 T)) and distribution and size of air voids enables the engineer to properly control the use of air-entraining agents. The density or unit weight of the concrete is used to measure the volume of a given batch of concrete and the proportions of ingredients of fresh concrete based on the volume of concrete produced [*2*]. This provides an accurate means of determining the cement content per cubic yard of concrete.

The number of tests of fresh concrete required will depend upon the uniformity of production. The concrete mix will generally be accepted, corrected, or rejected on the basis of individual tests. As in the case of aggregate acceptance tests, tests of fresh concrete may have limited statistical considerations since individual tests provide a basis for action. This does not preclude analysis of the uniformity and control of these properties.

In the final analysis, the strength of concrete measured by the strength of 6 by 12-in. cylinders broken in compression after 28 days of moist curing is generally accepted as one measure of concrete quality. Flexural strength of beams is also used extensively for pavements, and entrained air content is also required for concrete exposed to freezing and thawing.

It is assumed that samples taken each day represent the concrete placed during that day, and like other performance tests the acceptance of concrete is based on these tests. Due to the many factors that influence the strength of concrete and the variations that must be expected, individual tests are not reliable as a basis for action, and it is important that statistical consideration of the accuracy of an observed average be known. It is not within the scope of this paper to present a discussion of the limits of uncertainty of observed averages, but the reliability of standard tests for concrete will be mentioned.

The maximum error, for a given probability level, by which an average of given number of tests may differ from the true, unbiased average based on unlimited tests can be found as follows:

$$E = \frac{tV}{\sqrt{n}} \quad \text{..........(1)}$$

where:

E = maximum error of the average of the sample, per cent,

t = "Student's" t for $n - 1$ degrees of freedom at a specific probability level (Table 2),

V = coefficient of variation, per cent (Eq 5), and

n = number of tests.

Since most specifications for concrete require three 6 by 12-in. cylinders to be broken in compression at 28 days, the reliability of the average of such tests will be computed. Assuming a probability level of 90 per cent and the coefficient of variation among the three cylinders as 5 per cent, and substituting in Eq 1

$$E = \frac{2.920 \times 5}{\sqrt{3}} = 8.5 \text{ per cent..(2)}$$

In other words, for tests of three specimens alone, the error of the average will not exceed 8.5 per cent 90 per cent of the time. As the value of V becomes more reliable with an increased number of tests, the value of t is reduced, and if V can be established from a large number of tests, the value of t equals 1.645 for 90 per cent probability, and

$$E = \frac{1.645 \times 5}{\sqrt{3}} = 4.8 \text{ per cent..(3)}$$

It has been established by Committee 214 of the American Concrete Inst. [*3*] that for good control the coefficient of variation between companion specimens from the same batch should not exceed 5 per cent. It can be assumed then that where control is good, three cylinders will indicate the average strength of a batch of concrete with a maximum error of 4.8 per cent 90 per cent of the time.

TABLE 2—VALUES OF "STUDENT'S" *t* AT VARIOUS PROBABILITY LEVELS.

Number of Specimens − 1[a]	Probability Level				
	70	80	90	95	99
1.........	1.963	3.078	6.314	12.706	63.657
2.........	1.386	1.886	2.920	4.303	9.925
3.........	1.250	1.638	2.353	3.182	5.841
4.........	1.190	1.533	2.132	2.776	4.604
5.........	1.156	1.476	2.015	2.571	4.032
10.........	1.093	1.372	1.812	2.228	3.169
15.........	1.074	1.341	1.753	2.131	2.947
20.........	1.064	1.325	1.725	2.086	2.845
25.........	1.058	1.316	1.708	2.060	2.787
30.........	1.055	1.310	1.697	2.042	2.750
∞.........	1.036	1.282	1.645	1.960	2.576

[a] Degrees of freedom.

NOTE—Values of *t* originally presented by R. A. Fisher and F. Yates [*9*].

One batch of concrete does not necessarily represent all concrete for a structure or even the concrete produced in any day. Variations between batches are much greater than those within the batch, and for over-all variations, a coefficient of variation of 15 per cent can be expected for good control (Table 2). In establishing an average strength of concrete in a portion of a structure, three cylinders taken at different times with a coefficient of variation of 15 per cent would give an average whose error could be expected to be less than:

$$E = \frac{2.920 \times 15}{\sqrt{3}} = 25 \text{ per cent} \ldots (4)$$

E = 25 per cent 90 per cent of the time
Or if V is established from a large number of tests:

$$E = \frac{1.645 \times 15}{\sqrt{3}} = 14 \text{ per cent} \ldots (5)$$

The number of concrete tests that are required in general construction to provide a reliable estimate of the average can be estimated by using the established values for good control, that is, V equals 5 per cent for within-batch variation and 15 per cent for over-all variation. It can also be assumed that the error of average should not exceed 5 per cent 90 per cent of the time.

A reliable average for a batch of concrete will require:

$$n = \left(\frac{tV}{E}\right)^2 \ldots\ldots\ldots\ldots (6)$$

$$n = \left(\frac{1.645 \times 5}{5}\right)^2 \ldots\ldots (7)$$

n = 2.7 or 3 cylinders from one batch, and a reliable average for concrete produced from day to day will require:

$$n = \left(\frac{1.645 \times 15}{5}\right)^2 \ldots\ldots (8)$$

n = 24.4 or 25 cylinders from different batches.

According to these data, three cylinders are sufficient to provide a reliable estimate of a single batch of concrete, but because of the variations from batch to batch, one cylinder from each of 25 batches would be required to establish the average concrete strength represented by the 25 batches. More than one cylinder from each batch would not significantly increase the accuracy of the average. This illustrates the necessity of establishing the strength of concrete from the pattern of repeated tests.

These methods may also be applied in establishing the properties of fresh concrete and concrete aggregates but have limited significance except to determine the reliability of test methods. This can be accomplished by measuring the uniformity of repeated tests of the same material [*4*].

Construction Control Tests:

After the suitability of concrete and concrete aggregates is established, control tests made throughout construction will ensure production of uniform concrete of desired strength and quality. Control tests provide an excellent opportunity for the application of statistical methods, since it is possible to consider the pattern of test results over a period of time rather than individual samples, as in the case of acceptance and performance tests. As the control standard for a project or mixing plant is established, reliable estimates of the potential strength and uniformity of future production can be made.

Concrete control is generally established by the strength of 6 by 12-in. cylinders broken in compression after moist curing for 28 days. Tests of fresh concrete and certain aggregate tests, such as moisture content and gradation, may be considered control tests, but these are essentially check tests performed to maintain uniform slump, air content, grading, and moisture content. Such tests assist in the control of concrete but are not a measure of over-all concrete quality. Since all variations of materials and proportions are reflected in the strength of concrete, concrete control is established from uniformity of strength tests.

The usual objection to the use of 28-day strength tests is the fact that a great deal of poor concrete can be placed in a structure before test results are available. The reliability of the 28-day strength tests does not come from tests made the day concrete is placed but is based on the reliability of the project or mixing plant to produce good concrete, as established from previous tests. Once the reliability of control is established, it is possible to obtain a reasonable estimate of the future potential concrete quality. Variations from established control can be detected as additional test results become available. In some instances, particularly when important structural concrete is involved, it is desirable to include performance tests taken at the structure in addition to regular control tests. Until the reliability of control is established or as changes occur, control cylinders broken at 7 days or earlier are desirable in order to obtain early information on the potential strength of the concrete.

Steps in simplified computation of σ:
1. Round $\bar{X}$ to nearest 100 psi.
2. Divide deviations by 100.
3. Sample computations for data of Fig. 1.

Number of Tests With Same Deviation		Deviation Squared		Product
16	×	1^2	=	16
10	×	3^2	=	90
12	×	5^2	=	300
4	×	7^2	=	196
3	×	9^2	=	243
1	×	11^2	=	122
Sum 46				967

$$\sigma = \sqrt{\frac{967}{46}} \times 100 = 459$$

$$V = \frac{459}{3500} = 13.1\%$$

FIG. 1—Normal frequency distribution of strength data from 46 tests (taken from Ref. *3*).

Variations in tests of concrete on controlled projects can be assumed to fall into some pattern of the normal frequency distribution curve [*5*]. Where there is good control, test values are bunched close to the average, but if there are variations in test results, the values spread.

The amount of dispersion of test results is measured best by the standard deviation σ, which is the square root of the mean of the squared deviations of the individual tests from the average. The symbol σ generally denotes the standard deviations computed from large samples (theoretically when the number of samples equals infinity), and s denotes the standard deviation of small samples [*6*], although the difference is negligible when the number of tests is over 30. The method of computing the standard deviation is as follows:

$$\sigma = \sqrt{\frac{(X_1 - \bar{X})^2 + (X_2 - \bar{X})^2 + \cdots + (X_n - \bar{X})^2}{n}} \quad (9)$$

$$s = \sqrt{\frac{(X_1 - \bar{X})^2 + (X_2 - \bar{X})^2 + \cdots + (X_n - \bar{X})^2}{n - 1}} \quad (10)$$

where:

$X_1, X_2, \cdots X_n$ = individual tests
$\bar{X}$ = average of all tests, and
n = number of tests.

A simple, approximate method of computing σ, illustrated in Fig. 1, is practical and sufficiently accurate for concrete control.

The standard deviation expressed as a percentage of the average strength, $\bar{X}$, is called the coefficient of variation, V:

$$V = \frac{100\sigma}{\bar{X}} \quad (11)$$

and

$$V = \frac{100s}{\bar{X}} \quad (12)$$

Control tests, taken over a period of time, are superior in evaluating concrete quality because of the number of specimens involved. Tests taken each day for one month provide sufficient data so that the average and coefficient of variation can be used as a reliable basis for refining mix proportions, specifications, and design criteria [*3*].

The size of the project, types and amount of concrete produced, and the degree of control desired will dictate the number of specimens required. After concrete operations are progressing uniformly and initial adjustments of the mix have been made, samples of concrete taken each day for each type of concrete will provide a reliable pattern of uniformity. For large jobs, samples taken each shift will be desirable. Although one test cylinder from each sample will provide reliable information on the over-all variations of concrete strength over a period of time, companion cylinders from each sample will provide additional information on the "within-batch" or testing variations:

$$V_1 = \frac{100\bar{R}}{\bar{X}d_2} \quad \ldots\ldots\ldots (13)$$

where:

V_1 = within-batch coefficient of variation,

$\bar{R}$ = average range between companion specimens,

$\bar{X}$ = average strength, and

d_2 = a constant, depending on the number of companion specimens [*5*]. (For two companion cylinders $d_2 = 1.128$; for three companion cylinders $d_2 = 1.693$).

Within-batch variations are useful since it can be assumed that variations between companion specimens from the same sample are caused by discrepancies

TABLE 3—PROBABILITY OF LOW TESTS.[a]

$\frac{\bar{X} - f_c'}{\sigma}$	Probability of Tests Falling Below f_c'	$\frac{\bar{X} - f_c'}{\sigma}$	Probability of Tests Falling Below f_c'	$\frac{\bar{X} - f_c'}{\sigma}$	Probability of Test Falling Below f_c'
0.05	0.480	1.05	0.147	2.05	0.0200
0.10	0.462	1.10	0.136	2.10	0.0180
0.15	0.440	1.15	0.125	2.15	0.0160
0.20	0.421	1.20	0.115	2.20	0.0140
0.25	0.401	1.25	0.106	2.25	0.0120
0.30	0.382	1.30	0.097	2.30	0.0110
0.35	0.363	1.35	0.089	2.35	0.0090
0.40	0.345	1.40	0.081	2.40	0.0080
0.45	0.326	1.45	0.074	2.45	0.0070
0.50	0.309	1.50	0.067	2.50	0.0060
0.55	0.291	1.55	0.061	2.55	0.0050
0.60	0.274	1.60	0.055	2.60	0.0045
0.65	0.258	1.65	0.050	2.65	0.0040
0.70	0.242	1.70	0.045	2.70	0.0035
0.75	0.227	1.75	0.040	2.75	0.0030
0.80	0.212	1.80	0.036	2.80	0.0025
0.85	0.198	1.85	0.032	2.85	0.0020
0.90	0.184	1.90	0.029	2.90	0.0019
0.95	0.171	1.95	0.026	2.95	0.0016
1.00	0.159	2.00	0.023	3.00	0.0013

[a] *Sample Computation*

Data from Fig. 1:

$\bar{X}$ = 3500 psi,

f_c' = 3000 psi, and

σ = 462 psi.

Example:

To find the probability of strength tests falling below f_c' = 3000 psi

$$\frac{\bar{X} - f_c'}{\sigma} = \frac{3500 - 3000}{462} = 1.04$$

From Table 3, probability for 1.04 = 0.15 or 15 per cent. This means 15 tests out of 100 can be expected to fall below 3000 psi.

in making, handling, and testing the test cylinders [3].

Statistical methods provide tools necessary to prepare specifications based on the distribution of strength tests obtained from a structure. Committee 214 [3] recommends certain limitations based on the coefficient of variation. A more direct approach is to place limitations on the probability of tests falling below specification minimums. Table 3 is an adaptation of the table of the normal probability integral and provides a relationship between f_c', $\bar{X}$, σ, and the probability of low tests. The reliability of the probability figure is dependent upon the accuracy of the values for $\bar{X}$ and σ. The number of tests necessary to establish these statistics was previously discussed and will not change when used for this purpose.

TABLE 4—CORRELATION COEFFICIENTS AT THE 5 AND 1 PER CENT LEVELS OF SIGNIFICANCE.

Number of Tests − 2[a]	5 Per Cent	1 Per Cent	Number of Tests − 2	5 Per Cent	1 Per Cent
1	0.997	1.000	24	0.388	0.496
2	0.950	0.990	25	0.381	0.487
3	0.878	0.959	26	0.374	0.478
4	0.811	0.917	27	0.367	0.470
5	0.754	0.874	28	0.361	0.463
6	0.707	0.834	29	0.355	0.456
7	0.666	0.798	30	0.349	0.449
8	0.632	0.765	35	0.325	0.418
9	0.602	0.735	40	0.304	0.393
10	0.576	0.708	45	0.288	0.372
11	0.553	0.684	50	0.273	0.354
12	0.532	0.661	60	0.250	0.325
13	0.514	0.641	70	0.232	0.302
14	0.497	0.623	80	0.217	0.283
15	0.482	0.606	90	0.205	0.267
16	0.468	0.590	100	0.195	0.254
17	0.456	0.575	125	0.174	0.228
18	0.444	0.561	150	0.159	0.208
19	0.433	0.549	200	0.138	0.181
20	0.423	0.537	300	0.113	0.148
21	0.413	0.526	400	0.098	0.128
22	0.404	0.515	500	0.088	0.115
23	0.396	0.505	1 000	0.062	0.081

[a] Degrees of freedom.

NOTE—This table was taken from Snedecor [7]. Portions were originally from Fisher [8].

Special Investigations and Research:

Laboratory or field studies of concrete and concrete materials involving groups of tests to establish the relationship between variables do not involve a reliable estimate of the average as do acceptance and performance tests, nor do they involve uniformity of repeated tests as do control tests. The number of tests required will depend upon how apparent and pronounced the relationship is among the variables.

For example, the relation between concrete strength and cement content usually determined in trial mixes for mix proportioning can be estimated with relatively few tests, since the correlation between these variables is very high. If the correlation is not good, a few tests may not indicate the trend.

The relationship and correlation between variables can be established by computing the linear regression which represents a "best fit" of a straight line to the data [7]. If the relationship is obviously a curve, methods of computing curvilinear regressions can be found in most textbooks on statistical methods. Linear regression is the simplest relationship between two variables and will give a good approximation, in most cases, for a limited section of curve. The straight-line equation is computed as follows:

$$Y = \bar{Y} + \frac{\sum(XY) - n\bar{X}\bar{Y}}{\sum X^2 - n\bar{X}^2} \cdot (X - \bar{X}) \quad (14)$$

The correlation coefficient r, which is a measure of the degree of correlation between variables, is equal to:

$$r = \frac{\sum(XY) - n\bar{X}\bar{Y}}{\sqrt{(\sum X^2 - n\bar{X}^2)(\sum Y^2 - n\bar{Y}^2)}} \quad (15)$$

where:

Y = values of the dependent variable, plotted on the ordinate,

X = values of the independent variable, plotted on the abscissa,

$\bar{X}$ = mean of X values, and

$\bar{Y}$ = mean of Y values.

The independent variable is usually selected, such as cement content, and the dependent or unknown variable is established by tests, such as strength.

Table 4 indicates the significance of apparent relationships between two variables based on the number of tests involved. If the computed correlation coefficient is greater than the corresponding 1 per cent level of significance, the correlation is said to be highly significant, and if the correlation coefficient is greater than the 5 per cent level, the trend is significant. A correlation coefficient below the 5 per cent level is not considered significant. This does not mean there is proof of no correlation between variables, but it does indicate insufficient data to establish a trend. It is a warning that there is no reasonable assurance the sample is not from noncorrelated data [7].

SUMMARY

Tests of concrete and concrete-making materials will generally fall into the following general classifications: (*a*) acceptance and performance tests involving the reliability of individual tests and a reliable estimate of average, (*b*) construction control tests based on the uniformity of repeated tests, and (*c*) research conducted to establish relationships between variables. The majority of aggregate tests are acceptance and performance tests. Concrete tests are often adapted to performance, control, or research.

The size and number of samples will vary with the size of the job, quantity of concrete, maximum size of aggregate, funds and testing facilities available, and the accuracy desired. Large samples and numerous tests will increase accuracy but may not be practical.

Statistical methods provide valuable assistance in evaluating the quality and uniformity of concrete and concrete materials.

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THE QUALITY OF TESTING

By J. J. Waddell,[1] Personal Member, ASTM

Quality is defined as "an attribute, characteristic, or property" also "class, kind, or grade." In considering, then, the quality of testing, thought should be given to the characteristics of the kind of testing desired, how this kind of testing can be recognized, and how it can be obtained.

That testing of concrete and concrete materials is important and necessary has been demonstrated many times, especially when one reads of the spectacular structural failures caused by inferior materials. But there are other failures too, not so spectacular, but nevertheless serious and costly to the owner. Examples are popouts, cracking, and unsoundness of concrete resulting from the use of aggregates containing inferior constituents—constituents that would be revealed by proper tests, properly performed. Adequate quality control of materials, including laboratory and field tests, informs the engineer, architect, contractor, and owner of the properties of materials proposed for construction and serves as a guide to the producer in maintaining his product within specification limits.

There was a time, many years ago, when the engineer or architect depended entirely on his own experience in judging the quality of materials; the stick poked into an earth embankment, how far the concrete would flow in a form, and similar methods of evaluation. These methods are still good. Knowledge gained through experience is still, and always will be, a vital part of engineering proficiency. However, the engineer today finds it not only desirable but also necessary to rely upon laboratory tests to inform him of the suitability of a material. Different materials, new materials, and new usages for old materials all contribute to the need for reliable, impartial tests to point the way for correct use of these materials. This is especially true in the field of concrete construction where we see the development of bold and sophisticated structures that were not considered possible a few years ago. Ultimate strength design, thin shells, new lightweight materials, and exposed aggregate finishes are typical of modern trends in the use of concrete.

Whether we are concerned with setting up and controlling our own testing organization or purchasing the services from a commercial inspection agency, a discussion of testing is not complete without first some mention of samples and sampling methods. Abdun-Nur and Cordon, in their contributions have covered the theory and practice of sampling, discussing proper methods and pointing out the pitfalls to be avoided. Sampling has a pronounced effect on the quality of the test results, as a non-representative sample can be the basis for completely erroneous conclusions regarding quality of the material under consideration. Obviously the sampler has an obligation to both the supplier and the user, and this obligation is shared

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by the engineer who is responsible for the operation. Adequate instruction and supervision are necessary, and this implies supervisors who are at least aware of the statistical implications involved in any sampling plan, are qualified and empowered to make use of the proper procedures, and will see to it that they are followed.

Consideration should be given to the general relationships existing between the owner, architect, engineer, contractor, and testing agency. The "General Requirements" of the American Concrete Inst. (ACI) "Recommended Practice for Concrete Inspection (ACI 311-64)" are quoted here as being particularly appropriate. Substitution of "testing" for "inspection" makes them especially pertinent to this discussion.

> For the protection of the owner and the public, the responsibility for inspection should be vested in the architect or engineer as a continuing function of his design responsibility. The responsibility of the architect or engineer for inspection may be discharged by him directly, through his employees, or may be delegated to an inspection agency selected by the architect or engineer. In those cases in which the owner provides his own engineering service, the owner should select the inspection agency. The fee for inspection should be a separate and distinct item and should be paid by the owner directly to the architect or engineer or the inspection agency. Inasmuch as final responsibility for inspection rests with the architect or engineer, he should maintain close surveillance over whomever is carrying out the details of inspection for him. At no time should inspection or testing be made a function of the construction contractor. Furthermore, as a professional service, inspection should not be obtained on the basis of competitive bidding.

How, then, can the engineer know that the material has been tested properly? Besides the sampling just discussed, there are two main factors that enter here. First, does the selected test procedure give the required information, and second, was the test performed in accordance with the specified test method?

The first question is fairly easy to answer. Whatever the properties to be evaluated—strength, density, color, and so on—the laboratory report shows the values obtained in the test, expressed in certain units of measurement obtained by a certain method of test. If these units are the same as the ones specified and obtained in the specified manner, the user can then decide to what degree the material complies with the specified requirements. In this connection, it should be pointed out that the ACI Standard Building Code (ACI 318-63) of the American Concrete Inst. requires that "Tests of materials and of concrete shall be made in accordance with the standards of the American Society for Testing and Materials (ASTM), as noted elsewhere in this code." For this reason, all tests should be made exactly in accordance with the applicable ASTM method of test unless the client specifically instructs the laboratory to follow another method. Choice of the method of test is the responsibility of the person submitting the sample. If the laboratory does not follow exactly the ASTM or other specified method, the deviation and the reason therefore must be fully explained in the test report.

To be complete, the laboratory report should include comments relative to the appearance or behavior of the sample that could in any way affect its properties or value. For example, an obviously improperly made concrete cylinder strength specimen should be so reported. It is well to keep in mind the degree of accuracy required: in other words, the level of quality required by the particular test in order to give the necessary information. Unnecessary refinements should be avoided almost as much as carelessness. Specific gravity should be reported to the second decimal place, but reporting a sieve analysis to this apparent accuracy is an unnecessary refinement that adds nothing to the value of the data.

With the complete test report in hand, the engineer can now decide, on the basis of objective tests backed by his professional judgment, whether the material (*a*) complies fully with the specified requirements and may be used without reservation, (*b*) fails to comply and therefore cannot be used, or (*c*) falls in a borderline area of "substantial compliance" or "partial failure" in which it may or may not be used with certain reservations.

To determine whether or not a test has been performed properly in the manner as specified requires an evaluation of the entire laboratory plant. The criteria for this evaluation are: (1) qualifications and attitudes of personnel, (2) adequacy of plant and equipment, and (3) accuracy of test procedures. Adequacy in either plant or personnel can be offset by a deficiency in the other, leading to failure of the tests to measure up to the required standard of excellence.

Consider first the qualifications of the personnel. It is necessary to become informed of the qualifications not only of the operating employees but also of the supervisory staff. The supervisors are usually professional men and frequently of high standing in their field. These facts are easy to check. Consideration should be given to the reputation enjoyed by the principals of the firm. Their academic training is of importance, as well as the length and stature of professional and technical experience. Information of this type is available in the directories of the various technical, scientific, and professional societies, as well as in the brochures published by the laboratories themselves.

Similarly, the qualifications of the operating or working employees may be partially evaluated. However, these workers need not be graduates of engineering or scientific colleges, although such academic training is desirable. Usually they fall into two classes: (1) young university graduates obtaining experience to fit them for more responsible positions, or (2) qualified persons, limited in formal education, but possessing years of practical experience. Lack of higher education is not necessarily a reflection on the capability of these people. However, it is desirable that the laboratory management provide adequate training in laboratory techniques and related topics.

One factor of importance in judging the operating staff is the terms of employment. That is, are they hired on a when-and-as-needed basis, or are they permanent employees? Admittedly, testing of construction materials such as concrete is somewhat of a seasonal business in most parts of the country, requiring adjustments in the staff as the work load varies. However, a staff consisting largely of itinerant or temporary workers should be viewed with suspicion, except in those cases in which a temporary testing organization is set up for an especially large construction job.

Testing and evaluating materials is a team operation involving many persons and numerous steps. Every person in this team must be qualified to perform his part of the undertaking, keeping in mind the type of material and purpose of the testing. Each step is important, and departure from the specified method at any point is liable to cause questionable accuracy of the test results. Sampling, handling and preservation of the sample, preparation of the sample for testing, the actual execution of the test, and interpretation and reporting of test results must be performed by qualified personnel in accordance with the designated procedure if valid results are to be obtained. Other papers in this publication cover each of the steps in detail, and these papers should be the guide to assist the analyst in performing the work in accordance with the indicated ASTM or other test method.

A visit to the laboratory will usually suffice to evaluate the adequacy of plant and equipment. If the testing is being done within the engineer's own organization or company, he is usually in a position to control the quality of personnel and machinery made available for testing. If the testing is being done in a commercial testing or other type of laboratory, the owners usually take pride in showing their facilities. If they do not, there must be something wrong.

Whether or not the testing machines are in calibration is another matter. Completely erroneous results can be repeated with a good degree of reproducibility if the machine is not calibrated properly. For example, strength tests of concrete cylinders can be consistently high or consistently low if the testing machine is out of adjustment. Or a concrete air meter can give uniformly high or uniformly low results. Consideration of this point is closely interrelated with the final yardstick by which the accuracy of the tests is measured.

This is the most difficult part of the whole evaluation procedure and actually includes, at least partially, the other two criteria, qualifications of personnel and adequacy of the plant. Close adherence to the applicable ASTM standard methods of test is essential.

The use of standard reference samples is increasing in this respect. By performing a specified test on a standard sample possessing carefully controlled values, it is possible to arrive at an evaluation of the test procedure, including the calibration of the equipment. In this connection, the Cement and Concrete Reference Laboratory (CCRL) is doing much valuable work with individual laboratories in assisting them to evaluate their own operations.

How, then, is the engineer to judge the quality of testing offered to him? To answer this, he must use his own good judgement. There are, at present, no means by which he can arrive at a numerical value to grade the laboratory. A review of records kept by the laboratory on calibrations and checks of testing equipment would indicate, to some extent, whether maintenance of laboratory equipment has been systematic. The degree to which the laboratory conforms to the specified requirements, as reflected in the CCRL report, will be of great assistance in this evaluation. For this reason, a CCRL report should be demanded by the client or person ordering the tests. Armed with this information, the engineer can then evaluate the personnel, equipment, and procedures, as described herein, reaching a decision as to whether or not he considers the test results to be reliable and accurate.

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EVALUATION OF DATA

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Data are facts or figures from which conclusions can be inferred. The word carries with it connotations of objectivity, freedom from bias and personal prejudice, and an analytical approach in which precise measurement systems are applied to obtain information which in turn will be subjected to further analysis and interpretation leading to conclusions about the thing, group, or class of things to which the measurement system was applied. The long step between collection of information and conclusions is, to a large extent, evaluation of data. Furthermore, many of the considerations that should be applied to the proper evaluation of data should also be applied to the preceding steps in the operation. That is, planning the collection of data and the data collection itself.

Data evaluation can be simple, and it can be extremely complex. An examination of a simple problem and one of greater complexity will serve to illustrate several of the fundamental concepts.

In the simple case, suppose we have two blocks of concrete and the question is to decide which of the two is heavier. We also have available a scale of sufficient capacity that will weigh these blocks to the nearest pound. Accuracy and precision of the measurement system are not in question. We put Block A on the scale, and it weighs 83 lb; we put Block B on the scale, and it weighs 112 lb. These are the data. Evaluation of these data is to look at the two numbers and decide whether one is larger than the other. The analysis shows that the weight of Block B (112 lb) exceeds the weight of Block A (83 lb), and, therefore, we conclude that Block B is heavier than Block A. We know all there is to know, all that needs to be known in order to answer the original question with complete confidence. We do not have to say that *probably* Block B is heavier than Block A, or there are *indications* that Block B is heavier than Block A, or the trend of the data *seems to indicate* that it is *likely* that Block B is heavier than Block A. Block B *is* heavier than Block A and that's that.

Now let's turn to the more complex problem. In this case Mr. A and Mr. B each have a whole back yard full of concrete blocks, and the question to be answered is this. Is the average weight of the concrete blocks in Mr. A's back yard any different from the average weight of the concrete blocks in Mr. B's back yard? Since the back yards are fairly large and the concrete blocks fairly small, we are dealing with many thousands of units. Again we have the same scale available to weigh these blocks. It is accurate and precise to the nearest pound, and we assume we have someone who can read the scale correctly each time.

The question posed in this case can be

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answered with the same confidence, the same certainty as in the simple case. If we are willing to weigh each block in each back yard, add the weights, and divide by the number of blocks in each case and compute the average, these numbers can be compared and a conclusion can be reached. The main difference between the two situations is in the amount of work involved. In the latter case, we do not have single units to compare but two universes of units. The method described was to census each universe, to measure the characteristic of interest on every unit in each universe, and draw a conclusion on the basis of complete information. To do anything short of this is to introduce some measure of uncertainty in the conclusion, but obviously it may be completely uneconomical to seek absolute certainty. In fact, many times, if we made a measurement on every element in the universe, the result would be to destroy the very thing we are interested in.

The application of the mathematics of probability and statistics to sampling and evaluation of data provide the means whereby one can look at something less than an entire universe (a sample), make measurements on this subgroup of the whole, estimate the value that would be obtained if all elements were subjected to measurement, and attach to this estimate some numerical measure of confidence.

There are many definitions of statistics not all of which are complimentary. One was stated recently by William Kruskal, professor of statistics at the University of Chicago. He said, "Statistics is the study and informed application of methods for reaching conclusions about the world from fallible observations" [*1*].[2] Others have said that statistics is the science that is concerned with chance variation [*2*]. Statistical methods can be of use wherever conclusions are to be drawn or decisions made on the basis of experimental evidence. They can be used to summarize or describe data in such a way as to make them more meaningful and to generalize concerning a large amount of possible data from a smaller amount of available data. It is this second category or application that is of concern in this paper. These latter methods involve inferences or predictions based on a relatively small number of observations but applied to populations containing large masses of data that could be collected. For almost any attribute there will be a distribution of values for elements in the population. Absolute truth would be known only if every element in the population was examined, but in most cases one must be content with estimates of the truth. However, with the help of statistical inference, attached to these estimates one will have a measure of the probability of being wrong.

Basically there are generally four situations which are of concern:

1. Estimating population parameters from sample data, with a specified probability of being right (or wrong).
2. Comparing means or variances or both from two or more sets of data to determine if significant differences exist between or among the sets.
3. Providing a basis for accepting or rejecting a material.
4. Developing functional relationships among two or more variables.

These factors must be kept in mind for the end result desired dictates the approach and type of statistical analysis. It is, therefore, important that prior to sampling and testing to (*a*) decide what information is desired from the evaluation and (*b*) choose the evaluation procedure such that the necessary information can be obtained efficiently and

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

economically. Once these decisions have been made the type and amount of data required can be logically deduced, the sampling procedure can be selected, and the data obtained. Considerations of: Techniques, Procedures and Practices of Sampling; Size and Number of Samples and Statistical Considerations in Sampling; and Quality of Testing, have been presented in the first three papers. In this paper, the purpose is to introduce some basic statistical concepts and to show or give reference to how these and others are used in the four situations just enumerated.

DEFINITIONS

At this point several definitions must be introduced to permit logical development of later sections.

Measures of Central Trend:

Arithmetic Mean—A collection of numerical data will usually consist of a set of numbers that exhibit some variation; they will not all be the same. Frequently there will be a value around which the data tend to cluster, although this might not be obvious in all cases. The term average is frequently applied to this central value.

Many types of averages are in common use, but the one of concern here is the arithmetic mean. The arithmetic mean of a set of n numbers is the summation of the numbers divided by n.

Median—The median value of a set of n numbers, when n is odd, is the middle number in the set when the numbers are ranked in order of magnitude. If n is even, the median of the set is the arithmetic mean of the two middle numbers.

Mode—The mode is the most common value of a set, the number that occurs most often. It is a useful statistic in dealing with large sets of numbers but can be unreliable for small samples.

Measures of Variability:

Range—The range of a set of numbers is the maximum value minus the minimum value. It is simple to calculate, and by definition it gives to total extent of variation in the set. For small samples, it can be a useful statistic, but it is of practically no value for samples of say 8 or 10 or more. It has a prime defect; one or two exceptional cases can inflate the range and make it misleading as a measure of the variability.

Standard Deviation—The standard deviation or root-mean-square deviation of a set of numbers is the second measure of dispersion that is most commonly encountered. The standard deviation of a set of n measurements is found by summing the squared deviations from the arithmetic mean, dividing this sum by $n - 1$, then taking the square root. Let

σ = standard deviation,
n = number of measurements (numbers) $x_1, x_2 \cdots x_n$, and
$\bar{X}$ = arithmetic mean $= \dfrac{\sum x_i}{n}$.

Then

$$\sigma = \left(\sum_{i=1}^{n} \frac{(x_i - \bar{X})^2}{n - 1} \right)^{1/2} \quad \ldots\ldots (1)$$

For computing on a desk calculator, this equation can be put into the following form:

$$\sigma = \left(\frac{\sum x_i^2 - (\sum x_i)^2/n}{n - 1} \right)^{1/2} \quad \ldots (2)$$

Variance—Variance is defined as the square of the standard deviation.

Coefficient of Variation—Coefficient of variation, V is usually expressed as a percentage and is defined as:

$$V = \frac{\sigma}{\bar{X}} \times 100 \quad \ldots\ldots\ldots (3)$$

Basic Statistical Concepts

Universe, Population, and Sample:

The words universe and population were used in preceding sections without specific definition. Some authors use these terms synonymously, defining a population or universe "as any set of individuals (or objects) having some common observable characteristic," further stating, "The term population may refer either to the individuals measured or to the measurements themselves" [*3*]. We prefer and will use the definitions and distinctions made by Ostle [*4*]. He defines a universe as a specified group of objects and a population as the totality of all possible values (measurements or counts) of a particular characteristic for the specified group. The universe may be a collection of real objects or it may be an imaginary collection of objects. It may be finite or infinite. Obviously, a universe can have several populations associated with it. Hence, the universe can consist of 243 individuals who hold membership on ASTM Committee C-9 or all the 6 by 12-in. cylinders that could have been taken from a truckload of ready-mixed concrete. We are normally concerned with one or more characteristics of each element in a population. For the universe of C-9 members the population of interest might be the IQ of all members. For the universe of concrete cylinders the characteristics could well be compressive strength, durability in freezing and thawing, or air content; the collection of such measurements comprising populations.

Considering now only numerical measurements and not characteristics of the go-no-go type (for example, the sex of C-9 members), each population of values will have a mean, a standard deviation, a coefficient of variation, and so on. For these population values the prime designation is commonly used; thus, population standard deviation is σ', population mean is $\bar{X}'$, and so on. This is to distinguish these from sample values, the next important concept to be introduced.

"A sample is a part of a population selected according to some rule or plan" [*4*]. It is important to know when one is dealing with a sample as opposed to a population. It is also important to know or be able to define the population from which the sample has been drawn. We most often deal with samples and use sample values to give us information about the sampled population (that is, to estimate population values). For example, suppose several air-content tests are made on samples of concrete taken from a large batch of concrete. Presumably one is interested in the average air content and perhaps the variability in air content in the batch. The best estimates available then, if these are the data on hand, are $\bar{X}$, the sample average, to estimate $\bar{X}'$, the true or population value and σ, the sample standard deviation, to estimate σ', the population standard deviation. The universe in this case is all the samples of concrete of a particular size that can be obtained from the batch, and the population is the collection of results of air content tests made on each of the samples. Two things remain to consider: (1), does it make any difference how this sample is selected and (2), is there any way to know how close the estimates are to the right answer? The first question will be considered now and the second after the concept of normal distribution has been discussed.

Randomness:

Since generalizing from sample to population is one of the main objectives in data evaluation and since these inferences can have probability statements associated with them only if the laws of mathematical probability apply, it is necessary that random samples be selected from the population.

If a chance selection process is used to obtain samples wherein the probability of selection for every item in the population is known, the sample is called a probability sample; if the probabilities are equal, the sample is a random sample. Stated again, a random sample is one in which each element in the population has an equal chance of occurrence.

A simple random sample can be obtained by identifying all elements numerically, then using a table of random numbers to select the sample. Other types of random samples can be obtained [*5*]. The physical situation and knowledge of the characteristics of the population usually suggest the randomization technique. The need for attention to this aspect of sampling cannot be overemphasized, since all that follows is predicated on the basic assumption of randomness. There is no place in statistical inference for the so-called representative sample, perhaps more accurately called a subjective sample. It is entirely possible that in some cases an experienced person can look over the situation, scratch his head, squint and say "take the sample right here"—and be right on the mark. But do not call this statistical evaluation nor use the associated mathematics to defend the position.

Normal Distribution:

If one were to mix a batch of concrete of sufficient size to cast fifty 6 by 12-in. cylinders, then cure and break these at 28 days, it would come as no surprise to anyone to find that these fifty test results were not identical. There would be variation in the observed values, and if we plotted a histogram or frequency polygon for these data, we would have a graphical representation of the frequency distribution of the results. There are an infinite number of ways that such results might be distributed, a few that are common and one that approximates the underlying pattern of variation in this and in so many other cases so as to make it the most important distribution to consider in an elementary approach to evaluation of data. This is the normal distribution. "The general concept of a frequency distribution is at the heart of statistical reasoning. To think of a set of varying numbers as having come from some distribution is fundamental in solving statistical problems. Moreover, certain theoretical distributions form the basis of many of the most valuable statistical tools" [*2*].

If a population has a normal distribution, the distribution curve is symmetrical and bell-shaped, showing some tendency for the existence of a few cases at some distance from the mean in each direction. It is a two parameter curve, these being the mean, $\bar{X}'$ and the standard deviation, σ' or its square, the variance. The equation of the curve for n cases can be written,

$$f(X) = \frac{n}{\sigma'\sqrt{2\pi}}\, e^{-(X-\bar{X}')^2/2\sigma'^2} \quad \ldots (4)$$

where:

$f(X)$ = the frequency of occurrence of the observed value X,

σ' = the standard deviation of the population of n cases, and

$\bar{X}'$ = arithmetic mean.

The area under the curve as defined by Eq 4 can be shown to be n.

This appears to be an unfortunately complicated expression to explain the pattern of variation in many physical (and other) phenomena, but what it says is that (*a*) in this distribution, most of the values are not too far from the mean, and there are few to be found at either extreme and (*b*) the "spread" is measured by the standard deviation. In

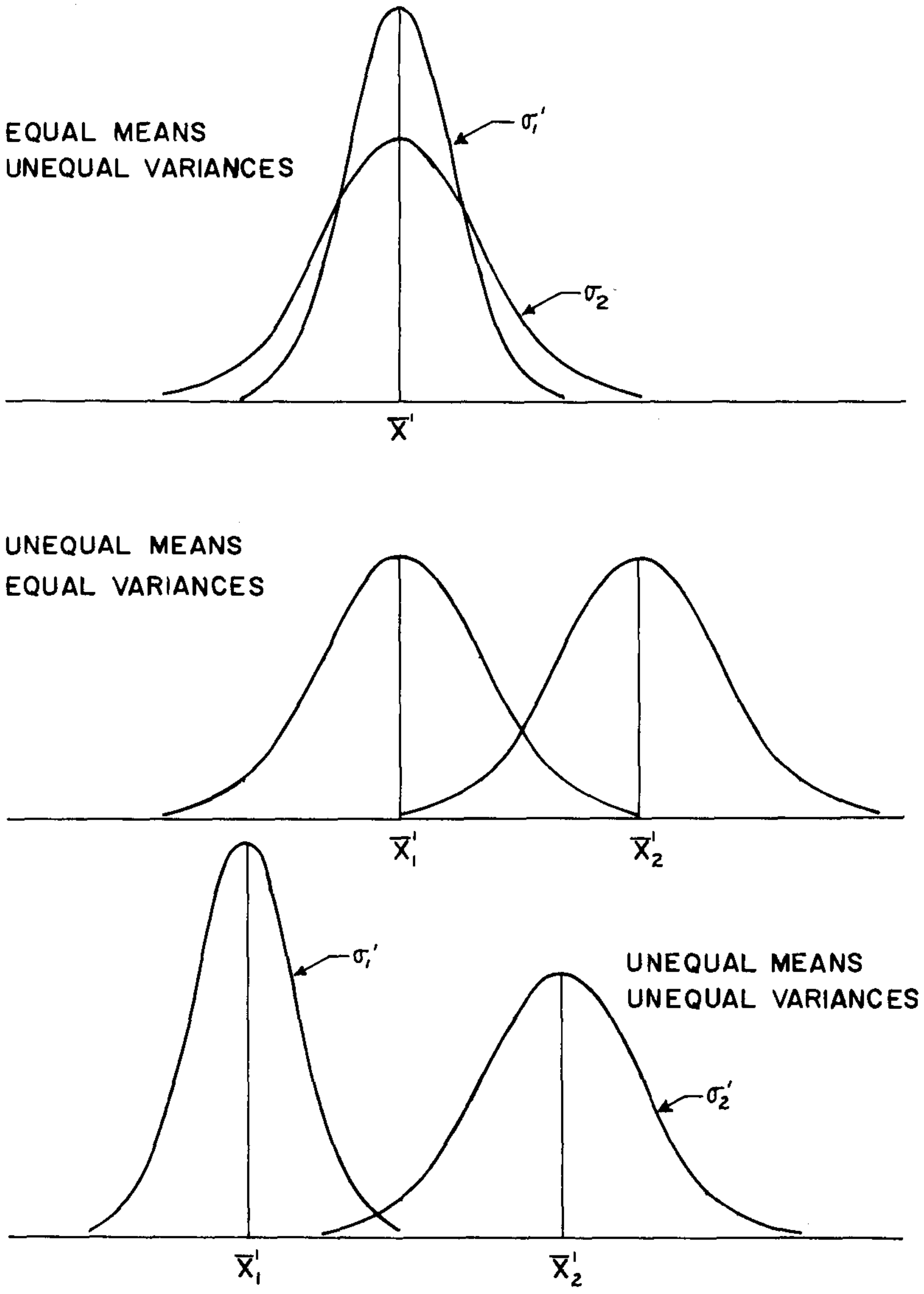

Fig. 1—Examples of normal curves.

addition, some manipulation of Eq 4 will show that the function has many desirable, useful properties. Examples of normal curves are shown in Fig. 1.

Equation 4 can be normalized (made free from physical units) by substituting the standard variable

$$Z = \frac{X - \bar{X}'}{\sigma'} \quad \text{.........} (5)$$

which may be interpreted as the number of standard deviations that X is above or below the mean (that is, $X = \bar{X}' +$

$Z\sigma'$). Then[3]

$$\phi(Z) = \frac{1}{2\pi} e^{-z^2/2} \ldots\ldots\ldots (6)$$

Figure 2 is a graph of the normal curve showing both X-unit and Z-unit scales. The following properties of the curve are stated without proof:

1. The maximum ordinate is at $Z = 0$ or $X = \bar{X}'$.

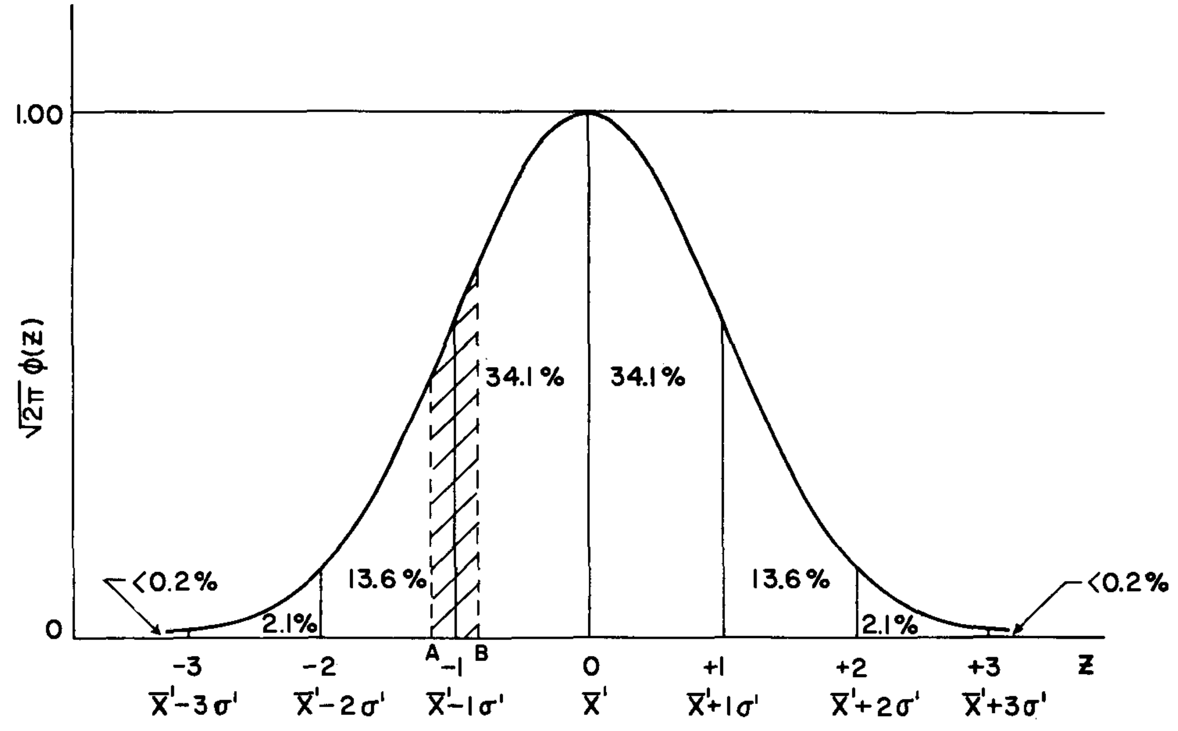

FIG. 2—Areas under the normal curve.

2. There is an axis of symmetry at $Z = 0$.

3. Points of inflection exist at $Z = \pm 1$ or $X = \pm 1\sigma'$.

4. The total area under the curve is 1.

5. The area under the curve between any two Points A and B is the probability of occurrence of a value in that interval.

6. The following is a tabulation of areas (probabilities) for intervals ±1, 2, and 3 σ' distant from the mean.

Interval	Area (probability)
$\bar{X}' \pm 1\,\sigma'$ ($-Z$ to $+Z$).........	0.6827
$\bar{X}' \pm 2\,\sigma'$ ($-2Z$ to $+2Z$).......	0.9545
$\bar{X}' \pm 3\,\sigma'$ ($-3Z$ to $+3Z$).......	0.9973

Area Under the Normal Curve—Areas under the normal curve above or below various values of Z have been tabulated. Such tabulations appear in many textbooks on statistics [*2,7*].

One can make good use of these known properties of the normal curve in problems related to concrete production. For example, suppose you were working to a three thousand psi minimum strength specification and you wanted to estimate what percentage of production could be expected to fall below this specification if operations were at some fixed level. Let's say, for example, that previous sampling and testing have shown that a certain mix design will produce an average strength of 3400 psi with a standard deviation of 340 psi. By making

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[3] In this derivation, Eq 4 is first made a probability function, $p(x)$, by dividing by n. The σ' in the denominator of the right-hand side drops out since $\phi\ (Z)dZ = p(x)dx$.

a simple calculation as follows:

$$Z = \frac{X - \bar{X}'}{\sigma'} = \frac{3000 - 3400}{340}$$

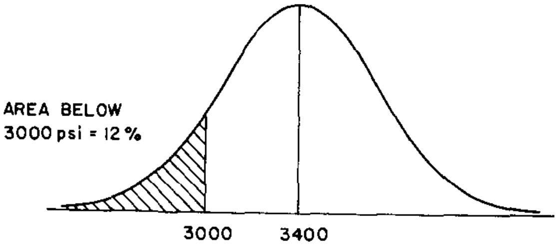
$$= -1.175$$

and going to a table of areas under the normal curve, one would find that the area under the normal curve below $Z = -1.175$ is 0.12 or 12 per cent. This means that 12 per cent of the sample results could be expected to lie below 3000 psi. A diagram of the situation should help illustrate this point.

TABLE 1—COMPARISON OF OBSERVED AND CALCULATED PERCENTAGES OF "LOW" TESTS FOR FOUR GROUPS OF DATA.[a]

	Data Group			
	1	2	3	4
Number of tests	406	181	688	458
Average strength, $\bar{X}$, psi	3400	3300	3630	3620
Standard deviation, σ, psi	515	557	450	426
Number of tests below 3000 psi	89	46	50	30
Percentage of tests below 3000 psi	21.9	25.4	7.3	6.6
Calculated percentage of tests below 3000 psi	21.9	29.6	8.0	7.5

[a] After Wagner, Ref. *6*.



If the concrete producer cannot live that dangerously, there are two alternatives: (*a*) operate at a higher average strength or (*b*) reduce the variability of the production, that is, reduce σ'. Numerically it might look something like this:

Case *a*—Increase average strength to 3700 psi. Then

$$Z = \frac{3000 - 3700}{340} = -2.06$$

and reference to the table would show that now less than 2 per cent of the samples can be expected to be below 3000 psi.

Case *b*—Maintain an average strength of 3400 psi but work hard on control and reduce the variability of the production. Say, for example the standard deviation was reduced to 270 psi, then

$$Z = \frac{3000 - 3400}{270} = -1.48$$

The area under the normal curve below -1.48 is about 0.07 or 7 per cent of the samples would, in the long run, be expected to lie below 3000 psi.

A comparison of this theoretical calculation with test results is found in a paper by Wagner [*6*] in which he reported the results of some 1700 compressive strength tests representing over 4300 individual concrete cylinders. The primary purpose of Wagner's investigation was to compare the effect of sampling and job curing procedures on compressive strength of concrete. In so doing he collected test data on cylinders made and handled by two types of personnel with maximum size aggregate as an additional variable. This resulted in four groups or classes of data. The results of interest here are summarized in Table 1. It can be seen that in all cases the percentage of test results below 3000 psi is in reasonable agreement with the percentage calculated from Eq 5.

TABLE 2—COMPRESSIVE STRENGTH TEST RESULTS.[a]

4270	3900	4680	4410	3750
4150	3500	3560	4320	4700
4360	4070	3700	4240	3580
4070	3770	2990	3640	4910
4860	3900	5020	3840	5030
3860	3040	3990	4040	3440
4210	3730	3970	4320	4570
4190	4050	4140	4450	3280
3830	4740	3080	3330	4490
3270	5000	3110	2960	3690

[a] Mean = 4000 psi; Standard deviation = 560 psi.

TABLE 3—COMPRESSIVE STRENGTH TEST RESULTS.[a]

4200	4150	4580	4210	3910
3850	4050	3840	3980	4350
4400	3990	4690	3810	3560
4320	4230	3900	4190	3770
3970	3680	4620	4270	4280
4020	4090	3860	4010	3790
3720	3950	3500	4100	4500
3650	3310	3800	4030	3420
4160	3620	4140	4290	3380
4380	4440	3710	3730	3600

[a] Mean = 4000 psi; Standard deviation = 330 psi.

TABLE 4—COMPRESSIVE STRENGTH TEST RESULTS.[a]

4260	4900	4820	5130	3700
4650	3400	4570	3720	3490
4960	4130	4360	4430	4210
3540	3660	4350	4410	4540
4080	3370	5360	4700	3910
5410	4250	4330	4710	4190
5440	5290	3990	4840	4080
5320	3830	5120	4500	4500
4560	4330	4760	3950	3380
4670	5070	4630	4160	4040

[a] Mean = 4400 psi; Standard deviation = 560 psi.

STATISTICAL INFERENCE

It is generally impractical, uneconomical, and often impossible to seek absolute knowledge of some characteristic of a universe by measurements on every element. Instead, a random sample of the population of interest is obtained, and from this the population parameters are estimated. Frequently we are interested in comparing results from two or more samples to decide whether or not it is reasonable to conclude that they are or are not from the same population. In this section, these problems are considered, and some of the basic tools for estimation and hypothesis testing are presented.

Test Data:

In order to have data on hand to illustrate the methods of estimation and hypothesis testing covered in this section, fictional test results were generated and are shown in Tables 2–4. Each table contains fifty numbers that represent the results of compressive strength tests on concrete cylinders. The numbers were contrived first to fit as close as possible to a normal distribution. Two of the sets (Tables 2 and 3) have equal means but unequal standard deviations. Two of the sets (Tables 2 and 4) have equal standard deviations but unequal means. The other possible combination (Tables 3 and 4) obviously have both unequal means and unequal standard deviations. These sets of numbers can be regarded and will be used in two different ways. Each can be regarded as a complete, finite population. That is, we can consider that each set of 50 cylinders constituted a universe and that each set of 50 strength test results is the population of strength values that gives complete information on that characteristic of the universe. Alternatively, each set of 50 numbers can be considered as a sample from some larger population.

Estimation:

A problem frequently encountered is to estimate the mean of a population from test results on a sample from the population. The best point estimate that can be made is the sample mean itself, since it can be shown that this statistic is unbiased, that is, the expected value of all sample means equals the population mean. However, the point estimate by itself gives no measure of the confidence that can be assigned to the result. Since sampling is involved, we know that certainty is out of the question, but it is possible to make an interval estimate to compute upper and lower limits that will include, with a specified probability of being wrong, the true mean. The interval so computed is called a confidence interval, the end points being termed confidence limits. The width of the band for a given estimate of a mean depends upon (*a*) how much of a chance one wants to take (confidence level), (*b*) the standard deviation and whether this value is known for the population or has to be estimated from the sample, and (*c*) the sample size. The two basic cases to consider are: (1) that in which the population standard deviation, σ', is known and (2) σ' is unknown.

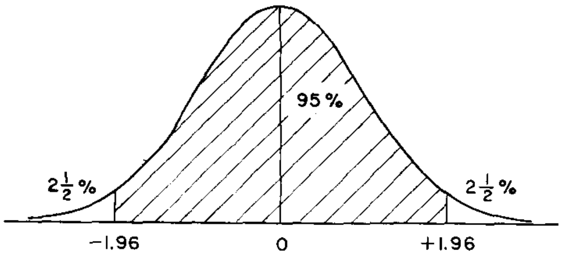
Interval Estimate, σ' Known—It can be shown that the sampling distribution of $\bar{X}$ has a mean $\bar{X}'$, a standard deviation $\sigma'/\sqrt{N}$ and is normal in shape. One can therefore use a table of areas under the normal curve, to establish the confidence interval for a sample mean. One has only to establish the confidence level, then compute the interval by the following expression:

$$\bar{X} \pm Z \frac{\sigma'}{\sqrt{N}} \cdots\cdots\cdots (7)$$

where:

N = number of cases in the sample,
$\bar{X}$ = sample mean,
σ' = population standard deviation, and
Z = tabular value for confidence level chosen.

The confidence level defines the area under the normal curve which, in the long run, will contain that percentage of the sample means. For example, if a 95 per cent confidence level is chosen, $Z = 1.96$, and the picture is as follows:



and we can say that with 95 per cent confidence the interval includes $\bar{X}'$, or that if 100 sample means based on N observations each are computed, and 100 confidence intervals computed, about 95 of these will include $\bar{X}'$. Other confidence levels can be chosen. For 99 per cent confidence, $Z = 2.58$ (99 per cent of the area lies between ± 2.58, ½ per cent above $+2.58$, ½ per cent below -2.58) and the interval widens. As was stated before, 100 per cent confidence—certainty, can be obtained only if every element in the population is known. Note also, this is the case where σ' is known—or at least can be assumed. This situation might arise where much information about a process had been collected over a long period of time.

Example 1:

The following sample of 9 was taken from the population of strength values shown in Table 2. Calculate the mean and place 95 per cent confidence limits on it.

3990	3080	4320
3970	3110	4240
4140	4410	3680

$\bar{X} = \Sigma X/N = 3882$
$\sigma' = 560$ (Table 2)

Confidence limits = $\bar{X} \pm 1.96\ 560/\sqrt{9}$
= 3882 ± 366 or 3516 and 4248

Interval Estimate, σ' Not Known—

More frequently the population standard deviation is unknown and the best estimate of this parameter available is σ, the sample standard deviation. In this case, the procedure is similar, but the sampling distribution is not normal and the "Student" t distribution must be used, the shape of which depends upon N, the number of observations or more precisely the degrees of freedom which in this case is $(N - 1)$. Intuitively we can say that if our estimate of σ' is based on a small sample, the confidence interval should be broader than if based on a larger one. It should be narrowest when the sample size is infinite, that is, σ' is known. Examination of a tabulation of the t distribution shows this to be the case; for infinite degrees of freedom and a percentile point of 97.5, corresponding to a confidence level of 95 per cent, $t = 1.96$ which is the Z value for 95 per cent confidence limits in the previous case. Examination of the preceding diagram shows why 97.5 is equivalent to 95 per cent confidence. The 97.5 defines the positive t value above which lies $2\frac{1}{2}$ per cent of the area. There is another $2\frac{1}{2}$ per cent below the corresponding $-t$ value.

Hence, to compute confidence limits for a mean in this case, use the expression

$$\bar{X} \pm t_{[(100+\text{CL})/2]N-1} \frac{\sigma}{\sqrt{N}} \quad \ldots (8)$$

where:

$t_{[(100+\text{CL})/2]N-1}$ = the tabular value of t for $(N - 1)$ degrees of freedom and a percentile point of

$$(100 + \text{Confidence Level})/2,$$

σ = sample standard deviation,
$\bar{X}$ = sample mean, and
N = sample size.

Example 2:

For the sample given in Example 1, compute 95 per cent confidence limits on the mean assuming σ' is unknown.

$\bar{X} = 3882$ from Example 1

$$\sigma = \left(\frac{\sum(X_i - \bar{X})^2}{N - 1}\right)^{1/2} = 496$$

Confidence limits $= 3882 \pm 2.26\ 496/\sqrt{9} = 3882 \pm 373$ or 3509 and 4255

Hypothesis Testing:

In the preceding section, attention was focused on the calculation of confidence limits on a sample statistic. Another, and perhaps more important problem, is one in which an assumption is made about a characteristic of one or more populations being sampled and this assumption is put to a quantitative test or examined in terms of its probability of being correct measured against some alternative assumption. The outcome of the test determines whether the hypothesis is accepted or rejected. This assumption is called a statistical hypothesis.

For example, one may have a sample of five results from tests made to estimate the air content of a nine cubic yard batch of concrete. We are interested in knowing whether the mean air content of the universe is equal to or greater than the lower specification limit on air content. The hypothesis is that the population mean $\bar{X}'$ is equal to or greater than some specified value $\bar{X}'_0$. The alternate hypothesis is $\bar{X}'$ is less than $\bar{X}'_0$, and the test of the hypothesis is the application of some quantitative rule that permits acceptance or rejection with a known probability of being in error.

Other examples of hypotheses that can be subjected to statistical test (paraphrasing Ref. *3*) are as follows:

1. This group of observations is a sample from a population with a mean equal to $\bar{X}'$:

(*a*) these concrete cylinders are from

production that has an average strength equal to the design strength,

(*b*) the average setting time for the concrete under these specified conditions is equal to some given number, and

(*c*) the average percentage of deleterious pieces in the aggregate is some given amount.

2. This group of observations is a sample from a population with variance σ'^2:

(*a*) the strength of concrete produced during June in Plant A is just as variable as the usual production and

(*b*) this mixer produces concrete of more uniform air content than the allowable variation.

3. These two groups are from populations with the same average ($\bar{X}'_1 = \bar{X}'_2$)

(*a*) Admixture A produces the same (or more, or less) air than Admixture B,

(*b*) concrete made with Aggregate A is more durable in freezing and thawing than concrete made with Aggregate B, and

(*c*) Product A is more effective in reducing water requirement in concrete than Product B.

Errors in Hypothesis Testing—As has been stressed in previous sections, if one makes inferences about populations from sample values, mistakes can be made. In hypothesis testing, two types of errors are possible.

Type I—Rejecting the hypothesis when it is really true.

Type II—Accepting the hypothesis when it is really false.

The probability of making a Type I error is called α.

The probability of making a Type II error is called β.

For a fixed sample size of N observations, one can choose α as he pleases but then, depending upon some specific alternative hypothesis being true if the original hypothesis is incorrect, β is fixed. "One of the objectives of hypotheses testing is to design a test whose α and β risks are both small. In most such test procedures α is set at some predetermined level, and the decision rule (when to accept the hypothesis or reject it) is then formulated in such a way as to minimize the other risk, β" [7].

Hicks [7] outlines in simple form a series of steps that apply to most types of hypotheses and test statistics. These are quoted below, following which stepwise examples are given for various types of tests that one might wish to make.

Steps in Hypothesis Testing (from Hicks, Ref. *7*):

1. Set up the hypothesis (H_0) and its alternative.
2. Set the significance level of the test, α. (Size of the Type I error).
3. Choose a test statistic to test H_0.
4. Determine the sampling distribution of this test statistic when K_0 is true.
5. Set up a critical region on this test statistic where H_0 will be rejected in (100)α per cent of the samples when H_0 is true.
6. Choose a random sample of N observations, compute the test statistic, and make a decision on H_0.

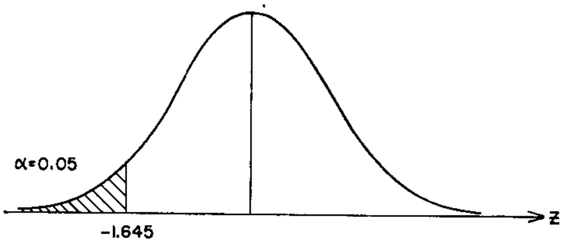
Example 3: (Population mean not less than some specified number, σ' known)

1. $H_0 : \bar{X}' = 4000$ psi
   $H_1 : \bar{X}' < 4000$ psi
2. $\alpha = 0.05$
3. Assuming σ' known = 560 psi
   Test statistic: $\bar{X}$ or standardized $\bar{X}$:

$$Z = \frac{\bar{X} - \bar{X}'}{\sigma'/\sqrt{N}}$$

4. $\bar{X}$ is normally distributed with a mean $\bar{X}'$ and standard deviation $\sigma'/\sqrt{N}$, or Z is normally distributed with a mean of zero and a standard deviation = 1 (see Fig. 2).

5. The critical region looks like



The critical region is $Z \leqq -1.645$; we reject H_0 if Z falls within it. This is an example of a "one-tailed" test, a single critical region at one end of the distribution curve.

The -1.645 is the Z value below which lies 5 per cent of the area under the normal curve and was obtained from tabulated values.

6. Let's use the same sample of nine strength results used in the previous example to test this hypothesis. From those data and from Table 2,

$N = 9$
$\bar{X} = 3882$
$\sigma = 560$

$$Z = \frac{3882 - 4000}{560/\sqrt{9}} = \frac{-118}{187} = -0.63$$

As $-0.63 > -1.645$, do *not* reject H_0.

Example 4: (Population mean not less than some specified number, σ' unknown)

If, in the foregoing example σ' was unknown, it would be estimated from the sample, and the applicable test statistic would be "students" t or

$$t = \frac{3882 - 4000}{496/\sqrt{9}} = \frac{-118}{165} = -0.72$$

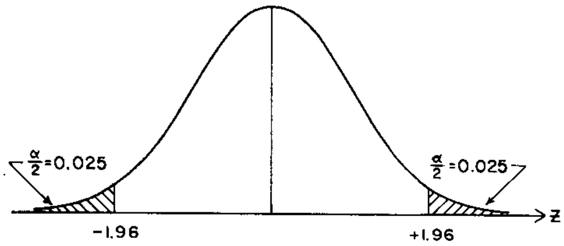
Reference to a table of t values for 8 degrees of freedom and 95 per cent (that is, 5 per cent of the area above or below), $t = \pm 1.86$.

As $-0.72 > -1.86$ again, do *not* reject H_0.

Example 5: (Population mean equals some given value, σ' known)

This case is used to illustrate the two-tailed test as distinct from the one-tailed test illustrated in Examples 3 and 4.

1. $H_0 : \bar{X}' = 4000$ psi
   $H_1 : \bar{X}' \neq 4000$ psi
2. $\alpha = 0.05$
3. Assume σ' known $= 560$ psi
4. $Z = (\bar{X} - \bar{X}')/(\sigma'/\sqrt{N})$, assume Z normally distributed, mean of zero, variance of unity.
5. In this case the critical region will look like the following:



The unshaded portion under the curve has an area equal to $1 - \alpha$ as before, but in this case the alternative hypothesis says we are equally interested in ruling out acceptance that $\bar{X}' = 4000$ when in truth it is greater than 4000 as we are in ruling out acceptance when the truth is that $\bar{X}' < 4000$. Therefore, the critical region consists of all values of Z below $Z_{\alpha/2}$ and above $Z_{1-\alpha/2}$. These regions are the area above $Z = +1.96$ and below $Z = -1.96$.

6. Taking the same numbers as used in Example 3,

$N = 9$
$\bar{X} = 3882$
$\sigma' = 560$

$$Z = \frac{3882 - 4000}{560/\sqrt{9}} = -0.63$$

As $-1.96 < -0.63 < +1.96$, do *not* reject H_0.

Example 6: (Population mean equals some given value, σ' not known)

If, in the preceding case, σ' is not known, the applicable test statistic is

$$t = \frac{\bar{X} - \bar{X}'}{\sigma/\sqrt{N}}$$

We assume this statistic has a t distribution with 8 degrees of freedom.

$$t = \frac{3882 - 4000}{496/\sqrt{9}} = -0.72$$

For a two-tailed test $\alpha = 0.05$ and 8 degrees of freedom, the critical regions are those above $t = +2.31$ and below $t = -2.31$

As $-2.31 < -0.72 < +2.31$, do *not* reject H_0.

Power:

Earlier, reference was made to α and β, the probabilities of making Type I and Type II errors, respectively. In the examples given, α was chosen as 0.05. It could just as well have been 0.01 or 0.10 if the experimenter decided that the consequences of rejecting a true hypothesis were more or less critical. Presumably the probability, β, of making a Type II error is also of interest to the experimenter—he wants to know the chances of accepting the hypothesis when some alternative hypothesis is really true. This is sometimes stated in terms of $1 - \beta$, the probability of rejecting the hypothesis if an alternate hypothesis is really true. Stated this way, $1 - \beta$ is called the power of the test; it is the probability that the test criterion will fall in the critical region if some alternate hypothesis is true.

A high power is desirable, one wants to reject H_0 if it is not true. Power depends on the α level chosen. For some given hypothesis and alternate, as α is made smaller, β increases, $1 - \beta$ decreases, power decreases. Power depends on the alternate hypothesis. For a given α, $1 - \beta$ increases as the alternate hypothesis becomes more remote from the hypothesis itself, that is, if the hypothesis is $\bar{X}' = 4000$ and the alternate hypothesis is that $\bar{X}'$ is different from 4000 by ±5000, the chances of accepting H_0 when this alternative is true are minute even for a sample of 9 and a standard deviation of 600. The power of a test may be increased by increasing α or by increasing the sample size.

A graphical representation of the power of a test is given by a power curve or alternatively by an operating characteristic curve. Ostle [*4*] states, "Since the power depends on the difference between the value of the parameter specified by the hypothesis and the actual value of the parameter where the latter is unknown, $1 - \beta$ should be expressed as a function of the true parameter. Such a function is known as a power function and is expressed as $1 - \beta(\theta)$ where θ represents the true parameter value. The complementary function $\beta(\theta)$, is known as the operating characteristic (OC) function."

Hypothesis testing, confidence limits, power, and so on are treated in detail in Refs. *3*, *4*, and *7* as well as in many other statistical texts.

Comparison of Two Means:

As a part of evaluation of data, many studies are concerned with determining if significant differences exist between the means of two sets of data. The objective is to determine if the two sets are from the same population, perhaps to decide if one process or product is superior to another. The hypothesis might be that the mean compressive strength of concrete cylinders molded in the afternoon equals that of cylinders molded in the morning. This statistical hypothesis, (H_0), may be expressed mathematically as $\bar{X}'_A = \bar{X}'_B$.

The alternate hypothesis for H_0 may be

$$H_1 : \bar{X}'_A \neq \bar{X}'_B \text{ or}$$
$$H_2 : \bar{X}'_A > \bar{X}'_B \text{ or}$$
$$H_3 : \bar{X}'_A < \bar{X}'_B$$

H_0 will be rejected if the test statistic falls in the critical region as before. The one-tailed test is characterized by H_2 and H_3 while H_1, is a two-tailed test.

Since the distribution of a sample is affected by the variance of the population, it is necessary that the variance be known or some assumption be made about it, if this type of comparison is to be made. There are three situations which may arise: (1) the population variances σ_A' and σ_B' are known; (2) the magnitude of σ_A' and σ_B' are unknown, but they are known to be or assumed to be equal; and (3) σ_A' and σ_B' are unknown and may not be equal. Assumption 2 is the one frequently made in the evaluation of concrete data. It is rather infrequent that the population variances are known (Case 1). Case 3 is not uncommon, and when it is suspected that σ_A' may not be equal to σ_B' then the test of means should be made accordingly. A test to determine if variances are equal can be made (Ref. *4*, p. 123).

For *Case 1* the following statistic is used to test the hypothesis that two means are equal.

$$Z = \frac{(\bar{X}_A - \bar{X}_B) - (\bar{X}'_A - \bar{X}'_B)}{(\sigma_A'^2/N_A + \sigma_B'/N_B)^{1/2}} \quad \text{..(9)}$$

This statistic will have a normal distribution with a zero mean and a unit standard deviation [*8*]. If the computed value of Z exceeds the Z value for the chosen α level, then the hypothesis that $\bar{X}'_A = \bar{X}'_B$ is rejected, if not it is accepted. "When the standard deviations of two universes are known, the test of a difference between two means is a normal test, regardless of sample size. For if the universes are normal, means of samples therefrom are normally distributed and likewise differences between them. Even if the universes are moderately nonnormal, means and differences of means will still tend to be normally distributed" [*8*].

To illustrate this procedure a sample of 5 values from Table 2 and 7 values from Table 3 will be used. Since these data are arranged in a random order the first five strength values from Table 2 and the first seven values from Table 3 will be used.

Data from Table 2	Data from Table 3
4270	4200
4150	3850
4360	4400
4070	4320
4860	3970
	4020
	3720

The hypothesis to be tested is $\bar{X}'_2 = \bar{X}'_3$. It is known that $\sigma_2' = 560$ and $\sigma_3' = 330$. If an α level of 0.05 is selected, then $Z_\alpha = 1.96$ for an alternate hypothesis that $\bar{X}'_2 \neq \bar{X}'_3$. Now, $\bar{X}_2 = 4342$ and $\bar{X}_3 = 4069$. Therefore,

$$Z = \frac{(4342 - 4069)}{\left(\frac{(560)^2}{5} + \frac{(330)^2}{7}\right)^{1/2}} = \frac{273}{278} \approx 1.0$$

which is less than 1.96 and the hypothesis is not rejected.

When *Case 2* is assumed, the t statistic is used for testing the difference between two means and the sample variances σ_2^2 and σ_3^2 are used in the calculation of t. "The t test is an exact test if the universes are normal and have a common variance. If they are moderately nonnormal but have common variances, the t test will give fairly good results" [*8*]. The t statistic is computed from the following equation:

$$t = \frac{(\bar{X}_A - \bar{X}_B) - (\bar{X}'_A - \bar{X}'_B)}{\sigma \left(\frac{1}{N_A} + \frac{1}{N_B}\right)^{1/2}} \quad \text{..(10)}$$

where:

$$\sigma = \left(\frac{N_A\sigma_A^2 + N_B\sigma_B^2}{N_A + N_B - 2}\right)^{1/2}$$

The value of t is calculated from Eq 10. An appropriate α level is chosen and the value of t_α or $t_{\alpha/2}$ obtained from tables of t values for $(N_A + N_B - 2)$ degrees of freedom. These tables are available in most statistical texts and in the ASTM Manual of Quality Control of Materials. If $t < t_\alpha$ (or $t_{\alpha/2}$ depending upon alternate hypothesis) then the hypothesis is accepted, if not, it is rejected.

This procedure may be illustrated by selecting ten strength values from Table 2 and ten values from Table 4. Assuming that the population variances are unknown but equal, the t statistic is calculated.

Data from Table 2	Data from Table 4
4270	4260
4150	4650
4360	4960
4070	3540
4860	4080
3860	5410
4210	5440
4190	5320
3830	4560
3270	4670

The hypothesis to be tested is $\bar{X}'_2 = \bar{X}'_4$. The computed values of $\bar{X}_2$ and $\bar{X}_4$ are 4107 and 4689, respectively, with $\sigma_2^2 = 168{,}512$ and $\sigma_4^2 = 383{,}232$. For the alternate hypothesis that $\bar{X}'_2 \neq \bar{X}'_4$, $t_{\alpha/2}$ is 2.10 for an α level of 0.05 and $(N_2 + N_4 - 2) = 18$ degrees of freedom. (If the alternate hypothesis was $\bar{X}'_2 < \bar{X}'_4$, t_α would be 1.73 for an α level of 0.05 and 18 degrees of freedom). Therefore,

$$t = \frac{582}{554\left(\frac{1}{10} + \frac{1}{10}\right)^{1/2}} = 4.70$$

which is greater than $t_{0.05} = 2.10$ and the hypothesis that $\bar{X}'_2 = \bar{X}'_4$ is rejected.

If the variances are unknown and there is doubt that they are equal, *Case 3,* then the test becomes somewhat more complex. The value of t is computed from the following equation:

$$t = \frac{(\bar{X}_A - \bar{X}_B) - (\bar{X}'_A - \bar{X}'_B)}{\left(\frac{\sigma_A^2}{N_A} + \frac{\sigma_B^2}{N_B}\right)^{1/2}} \quad \text{..(11)}$$

The value of t_α depends on the chosen α level and degrees of freedom computed by the following equation:

$$df = \frac{1}{\frac{c^2}{N_A - 1} + \frac{(1-c)^2}{N_B - 1}} \quad \text{..(12)}$$

where:

$$c = \frac{\sigma_A^2/N_A}{\sigma_A^2/N_A + \sigma_B^2/N_B}$$

This procedure is known as the Aspen-Welch Test (*Biometrika,* Vol. XXXVI, 1949, p. 290).

Analysis of Variance

In the analysis of data by statistical methods, one of the most powerful statistical tools is the analysis of variance, frequently referred to as the ANOVA. The ANOVA as a statistical tool allows the simultaneous study of the variation among several sample means consideriing all elements that contribute to the total variance. Most of the basic statistical information for significance tests can be obtained from the ANOVA. An analysis of variance may be used to make decisions as to whether variation in a measured characteristic is real (assignable to a recognized variable) or due to chance. Of the ANOVA, it has been said that "Basically it consists of classifying and cross-classifying statistical results and testing whether the means of a specified classification differ significantly" [*8*].

Other statistical techniques, such as hypothesis testing and estimates of population parameters, are a part of the analysis of variance. The statistical results of an analysis of variance may be used to set confidence limits, test hypotheses, determine control limits, estimate variances, and so on. The technique of analysis of variance can be used in situations where the final measurement can be controlled at a series of fixed levels or subdivided into classes [*9*]. It should be noted that when other statistical techniques are used in conjunction with an ANOVA the basic assumptions for each technique must still be met as well as the additional assumptions for the ANOVA.

Consideration of ANOVA should not begin after the data have been collected but before. To accomplish a stated objective there are, generally speaking, many experimental designs that could be used. The choice of the model must be made by the experimenter, but the choice and resulting analysis will be influenced by the information sought. The simpler the model the better. A complicated model is a waste of time and effort, if the same results can be achieved with a simpler one. The choice of a proper design of the experiment is a primary prerequisite for a statistical analysis that will give the desired information.

Numerical examples of the application of the analysis of variance to even a simple example would require more space than is warranted in this treatment of the subject. Excellent coverage of the subject can be found in Refs. *3*, *4*, and *7* and in many other statistical texts.

CONTROL CHARTS

The control chart became a well established technique in production quality control during the World War II era. The control chart is a combination of both graphical and analytical procedures. The basis of the theory arises from the fact that the variation of a process may be divided into two general categories. One portion of the variation can be described as random or chance variation of the process and the other as the variation due to assignable causes. A process that is operating with only chance variation should result in some distribution of the measured parameter, and one should be able to predict a range within which a certain percentage of the data should fall. If some assignable cause (such as an increased water-cement ratio) results in a change in the distribution, then the values of the measured parameter could fall outside the predicted range.

Control charts are particularly useful in controlling the quality of a product manufactured with tools or dies which wear with time. As these tools or dies wear the variation in size of the product will increase. A control chart will graphically show the trend, and, when the tolerance level is reached, the machine may be retooled. Once the limits for a process have been established, little is left but to plot the data on the control chart and be ready to take corrective action when the data exceed the control limits.

Three types of control charts which are frequently used are control charts for averages, control charts for standard deviations, and control charts for ranges. Also, control charts for other measures, such as per cent defective, may be useful. Detailed treatment of this subject and tables of control chart constants for determining upper and lower control limits are presented in texts on statistical quality control [*2,8,10*]. Table B-2 of the ASTM Manual on Quality Control of Materials, Special Technical Publication 15-C, contains the control chart constants for averages, standard deviation, and ranges.

DEVELOPING FUNCTIONAL RELATIONSHIPS AMONG TWO OR MORE VARIABLES

The fourth situation of interest concerns seeking ways of expressing the form of a functional relationship between two or more related variables and putting some numerical measure on the strength of the association. These end points are accomplished by techniques of regression and correlation. "Regression methods are those used to determine the 'best' functional relation among the variables, while correlation methods are used to measure the degree to which different variables are associated [*4*]."

A typical picture that frequently comes to mind is that of a plot of two variables with a curve drawn through the data scatter. The regression analysis, using various techniques of curve fitting, will result in the equation of the curve. The functional relationship between the variable is thus defined. "In general, a regression problem considers the frequency distribution of one (or more) variable(s) when another is held fixed [*3*]." If three variables or factors are being considered, the problem is more complex but still can be graphically represented. When more than three variables are present, a graphical representation is no longer possible, but the problem can still be analyzed mathematically, although the labor involved is increased. Computers greatly facilitate the solution of multivariate problems.

In a correlation analysis the most frequent index used to express the degree of association is the correlation coefficient. This coefficient is defined such that it is unity if all points fit exactly some preselected function and zero if the points are randomly distributed. While the regression analysis will define a curve that will best fit the data, the correlation analysis shows how close the data come to the curve.

Regression analysis is applicable to problems where it is desired to know how a certain parameter, such as strength, varies with factors, such as aggregate gradation, water-cement ratio, and curing temperature. A correlation analysis will indicate how well the data are grouped to fit the regression curve.

As in other statistical methods (for example, analysis of variance) statistical techniques, such as hypothesis testing and determination of confidence limits, are frequently used to supplement regression and correlation analysis. Examples of this would be in testing the hypothesis that an observed slope is equal to the predicted slope and placing confidence limits on the regression curve.

The procedures for analysis of even a two variable system by regression and correlation methods are too lengthy to present here. References *3*, *4*, and *8* are suggested for study.

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NEEDED RESEARCH

By A. Allan Bates,[1] Personal Member, ASTM

> "Research . . . is essentially a state of mind . . . a welcoming attitude toward change."
>
> *Charles F. Kettering*

Definitions

"Concrete," from "*concretus*," past participle of the latin verb *concrescere*, meaning "to join together" or "to unite in growth." These definitions from Webster's Unabridged Dictionary emphasize that "concrete" is essentially a verb-form, not a noun, and that the word denotes a method or an operation, not a substance. It defines a process rather than a material. Or, more informatively, concrete is a mass of materials characterized principally by the manner in which they are united to form a functional composite. The materials involved are as diverse and numerous as may be required to serve the designer's ultimate purpose. These materials are not categorically limited to any particular classes, species, or forms.

The common characteristic of all concreted masses lies in the presence of a continuous matrix, or "cement," which binds together all of the individually discrete constituents. Currently, in building and construction, this cement, for economic reasons, is usually composed largely of an intimate combination of hydrated alkyline and alkaloidal alumino-silicates which may be modified, extended, or replaced chemically or physically, either unavoidably or in some desirable degree, by an unlimited variety of other substances. The cement performs its functions principally by means of complex adhesive and cohesive forces which are not well understood.

Research Unlimited

This publication concerns ". . . the Properties of Concrete and Concrete-Making Materials . . . ." Since "concrete making materials" now potentially comprise practically all the materials known to man, the body of research covering the subject can be vast indeed. Furthermore, since the properties of any given material *in* concrete may be quite different from the properties of that same material considered independently, the "needed research" has no discernible limit. And still further, since concretes are of infinite variety, the properties of any material or combination of materials "in concrete" are likewise of potentially infinite variability.

Any investigator familiar with concreted materials and structures can go on and add innumerable circumstances and conditions which affect the properties of the system; as examples, prior history of component substances, states of stress, temperature and temperature cycles, contact with fluid ambients both constant and varying, incidence of radiation, and so on. Therefore, the sum total of fact and possible knowledge about "the properties of concrete and concrete-

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making materials" is beyond attainment and probably beyond necessity. Thus, "needed research" is that required by an individual or organization to meet a particular set of circumstances at a given time and place; this research will involve both generalized aspects more or less well known to every competent investigator and special problems pertinent only to a given structure and definable in advance only by an experienced and highly trained person. Such persons will never be available in adequate number.

The "1965" Compilation of Research in Progress in Plain and Reinforced Concrete" published by the American Concrete Inst. lists over 1800 projects under way in 278 laboratories in some 25 countries. This includes perhaps not more than half of all the projects in actual progress in the world, since a great many countries in which extensive concrete research is carried on are not listed and many laboratories even in the listed countries are not included.

These thousands of current research studies in concrete going on all over the world emphasize several important points, for example: (1) in the field of building and construction concrete technology is uniquely universal, (2) a comprehension of "needed research" in concrete must be based upon knowledge of what is being done in the other nations of the world, (3) the "need" which might most usefully be fulfilled is for a world-wide system for promptly exchanging, abstracting, and evaluating the research in progress on concreted materials and structures everywhere.

There are several organizations in the United States and elsewhere which undertake in a geographically limited or technically partial manner to provide the information system suggested above. Indeed, this American Society for Testing and Materials' report is of that nature. Likewise, the American Concrete Institute Compilation of Research in Progress in Plain and Reinforced Concrete is a step in the indicated direction. In Europe such organizations as CIB (International Council for Building Research Studies and Documentation) and RILEM (International Union of Testing and Research Laboratories for Materials and Structures) are also providing information services which include documentation on concrete research. The USSR also operates an extensive national technical abstracting bureau which no doubt gives much attention to concretes. Collectively, these and other similar existing organizations probably possess the capacity to keep research workers in concrete adequately abreast of the tide of new information being inspired by the current phenomenal world-wide activity in building and construction.

It can be confidently expected that there will be an eventual coordination of all these organizations into a universal research informational system using advanced mechanisms of classification, evaluation, and retrieval. The cost, though great, will not be excessive if equitably shared. And the advantages to the technology of concretes will be commensurately large. Leadership will no doubt develop in several centers among which we may hope that U.S. organizations will not be found lagging.

RESEARCH IN PARTICULAR

The opening paragraphs of this chapter suggest that we are entering a stage in the history of concretes in which the most fruitful research may result from a new "state of mind," a release from former limits of attitude. The Romans discovered the possibilities of concrete when some inventive building contractor mixed cheap, fine volcanic ash with the expensive lime mortar he was using.

Quite possibly he did this to lower his costs, but, as a by-product, he made possible the construction of the Roman Empire. In the phraseology of this report (see p. 543) he introduced a "Mineral Admixture." More realistically he invented "cementum." If he did this as a result of some research work, then he alone justified all of the research effort which has been expended on concretes and cements ever since. The profits are still coming in by the billions yearly.

There are certain broad and readily indicated areas in concrete technology which are particularly rich in opportunities for fruitful research. These areas result from fundamental economic, technical, and social trends which are either already in operation or fairly predictable, such as industrialization of building procedures, urbanization, rise in standards, and change in modes of human life, among others. For example, increasingly urbanized conditions require construction on crowded sites where storage of materials is difficult or impossible; hence, the pressure is great to accomplish as much as possible in off-site factories—of which the ready-mix truck is one. Production of building elements in factories puts a premium on quality control, speed of production procedures, high efficiency of design, output of finished products ready for installation (complete walls, and so on), on lightness (for transport to building sites), and others. Perceptive research organizations and workers on studying such considerations will readily find numerous technical problems and ideas on which to work.

The availability of natural aggregates is diminishing relative to demand and thus presenting growing problems. Study of ways to produce synthetic aggregates varying in weight, insulating quality, and aesthetic appeal will pay large dividends. Concrete made with such aggregates will present special problems.

Increasing prefabrication of structural units and building elements requires cements and other raw materials of high uniformity and reliability. Experience already acquired in other countries indicates that cements, aggregates, and admixtures of greater variety in terms of performance are needed. Performance during the production cycle, as well as in final building use, is being measured with increasing rigor. Some of the resulting demands for better performance can be met by already available additions and admixtures to normal concrete. However, the possibilities for increasing the versatility of concretes by the use of admixtures provides a research field of literally unlimited extent.

Concrete, like all other materials used in building, is being increasingly used in composite design which places exacting requirements for physical and chemical compatibility on the several materials which are intimately in contact. Some unfortunate experiences have occurred as a result of lack of information in this field.

Before the next edition of this report comes off the press an immense amount of additional "needed research" will have been accomplished, but the needs for research will not have been diminished. Those needs, at least the profounder ones, are established more surely by the mental processes going on in the minds of research workers than by the exigencies of the market place or of the physical world.

PART II

Tests and Properties of Concrete

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THE NATURE OF CONCRETE

T. C. Powers[1]

The term concrete can be construed to include a considerable variety of products made from portland cement or other cementing media, but in this publication the term concrete usually refers to a material which was at first a plastic mixture (or mixture that became plastic as a result of manipulation, especially vibration) of portland cement, water, air, and mineral aggregate. Therefore, this discussion of the nature of concrete will have the scope indicated by that description.

A writer's concept of the nature of concrete can hardly be revealed in a few words, but his treatment of certain topics and his definitions are indicative. Here are some examples: in 1878 Trautwine, in the 11th edition of his *Pocket Book for Civil Engineers*, said "Cement concrete, or beton, is . . . cement mortar mixed with gravel or broken stone, brick, oyster shells, etc., or with all together." He described mortar as sand containing a volume of cement equal to the volume of voids in the sand.[2] In 1907, L. C. Sabin in a book on concrete said, "Concrete is simply a class of masonry in which the stones are small and of irregular shape. The strength of concrete depends largely on the strength of the mortar; in fact, this dependence will be much closer than in the case of other classes of masonry, since it may be stated as a general rule, the larger and more perfectly cut are the stone, the less will the strength of the masonry depend on the strength of the mortar." Feret in 1896 considered water and air to be definite components of mortar (and presumably also of concrete), but it is not clear that he thought of cement paste as an entity. Zielenski, once head of the Hungarian Association for Testing Materials, in 1910 called concrete a conglomerate body; he considered the conglomerate to be composed of mortar and coarse aggregate, and the mortar to be composed of paste and sand, with or without air voids. Taylor and Thompson, authors of perhaps the best of early books on concrete, in the 1912 edition said, "Concrete is an artificial stone made by mixing cement, or some similar material which after mixing with water will set or harden so as to adhere to inert material, and an aggregate composed of hard, inert, materials of varying size, such as a combination of sand or broken stone screenings, with gravel, broken stone, cinders, broken brick, or other coarse material." D. A. Abrams, in the first bulletin from the Structural Materials Research Laboratory, Lewis Inst., Chicago, Ill. (1918) emphasized the significance of the ratio of water to cement in concrete, and he abandoned the notion that concrete is a mixture of mortar and coarse aggregate, pointing out that the whole aggregate, fine and coarse combined, should be considered as one, even though fine and coarse aggre-

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[2] He said also, "Nearly all the scientific principles which constitute the foundation of civil engineering are susceptible of complete and satisfactory explanation to any person who *really* possesses only so much elementary knowledge of arithmetic and natural philosophy as is *supposed* to be taught to boys of 12 or 14 in our public schools."

gates are proportioned separately. F. R. McMillan in his book, *Basic Principles of Concrete Making* (1929) said, "Expressed in the simplest terms, concrete is a mass of aggregates held together by a hardened paste of portland cement and water . . . the paste is the active element." In their textbook on concrete, Troxell and Davis (1956) wrote, "Concrete is a composite material which consists essentially of a binding medium within which are embedded particles or fragments of a relatively inert mineral filler. In portland cement concrete the binder or matrix, either in the plastic or in the hardened state, is a combination of portland cement and water." This definition was adopted by the American Concrete Inst. (ACI) Committee 116 on Nomenclature in 1964. The Encyclopedia Britannica, 1963 edition, says, "Concrete is a building material consisting of a mixture in which a paste of portland cement and water binds inert aggregates into a rock-like mass as the paste hardens through chemical reaction of cement with water."

Although it is possible to discern an evolution of concepts in the above definition and descriptions, the early concept of concrete as a mixture of mortar and coarse aggregate tends to persist, despite Abrams' contention that the total aggregate functions as a unit. The idea lingers with us, perhaps, because it has simplicity and plausibility and partly because it is not altogether unrealistic, especially when there is a gap between the largest size in the sand and the smallest size in the coarse aggregate. However, gap gradings are not common, and in any case the correctness of Abrams' conclusion can hardly be questioned.

Even the latest definitions provide but a superficial idea of the nature of concrete. They give no hint as to how and why the originally plastic mass becomes hard and strong and, indeed, say nothing as to how and why the mixture had plasticity in the first place. Moreover, they give no adequate basis for understanding such aspects of concrete as volume change characteristics, and stress-strain-time phenomena. We must, therefore, go far beyond a superficial definition or description to attain today's understanding of the nature of concrete. To do the subject full justice would be a book-length project; I cannot do more than touch on a few fundamental topics.

GROSS STRUCTURE OF CONCRETE

When we inquire into the nature of concrete we find it necessary to regard concrete not as an entity—a substance—but as a structure having component parts, as has already been indicated. The predominant component of concrete is an aggregation of mineral particles, called the aggregate, and this aggregation requires a certain minimum of space per unit weight of material. The volume of space occupied by a properly compacted fresh concrete mixture is slightly greater than would be the compacted volume of the aggregate it contains, and the difference is significant: it shows that the individual rock[3] particles in concrete need not be in contact with each other. There is clear evidence that the rock particles in concrete are, in fact, not in contact with each other: freshly mixed concrete could not be plastic if the solid particles were not dispersed to some degree; inspection of broken sections of hardened concrete show that not only are the rock particles in a dispersed state while the mixture is fresh, but also they remain dispersed, although generally not exactly to the same degree that prevails immediately after mixing, owing to settlement under the force of gravity before setting occurs.

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[3] Throughout this discussion, the term rock refers to the particulate mineral matter that makes up the aggregate, regardless of the size or shape of the particles.

Rock particles in plastic concrete are dispersed in a matrix composed of paste and air bubbles;[4] the paste is composed of portland cement and water. The degree of dispersion actually depends upon the consistency of the paste and the volume of air; the stiffer the consistency and the higher the air content the greater the mean clear distance between aggregate particles. In practical terms, this means that the degree of dispersion of rock particles is greater the lower the water-cement ratio of the paste, and the higher the air content; in any case, the volume of concrete seldom if ever exceeds the compacted volume of the aggregate by more than 10 per cent, and usually it does not exceed it by more than 3 per cent when no air-entraining agent is used.

Without an air-entraining agent, concrete placed by a standardized procedure contains a characteristic amount of air in the form of bubbles, the amount being a function of certain variable factors. The main factors are the consistency of the paste and the gradation of the aggregate. At a given paste consistency, and with a standardized mixing procedure, air content depends mainly on those features of aggregate grading that control the mean size of the voids in the aggregate, voids being here defined as the space occupied by paste and air. The mean size and the size range of the air bubbles in concrete are also significant structural features, particularly with respect to the ability of concrete to withstand the effects of freezing. These characteristics also are subject to systematic variation as will be discussed later.

The foregoing statement as to the effect of aggregate grading on air content is applicable principally to the leaner types of mixtures. In richer mixtures, water-cement ratio less than about 0.5 by weight or $5\frac{1}{2}$ gal per sack, aggregate characteristics have little effect. Throughout the range of the most frequently used mixtures, air content and void characteristics are strongly influenced by water-cement ratio, the air content and average air void size increasing with increase of water-cement ratio under given conditions.

When a suitable air-entraining agent is used, the air content can be raised to almost any desired level. At an air content higher than that normally present in a given mixture, the mean size of air bubbles is controlled by the characteristics of the air-entraining agent used and, in the leaner range of mixtures, by the same factors that control the normal air voids. Specifically, if the normal air content of a given mixture is relatively high and the mean size of the voids relatively large (the two usually go together), raising the air content by means of a given approved air-entraining agent will result in a system of voids having a relatively large mean size, as compared with the mean size when the amount of air normally present is smaller, and the original mean size also smaller. This means that with a given air-entraining agent used in different mixtures a wide range in average void size may be observed.

In short, the gross structure of concrete appears to be that of an aggregation of rock particles slightly dispersed in a matrix of paste and air bubbles, the proportion and size characteristics of air bubbles being subject to systematic variation just as is the proportion of aggregate.

A conclusion arising directly from consideration of the gross structure of concrete is that the firmness or mechanical stability of concrete cannot be attributed to mechanical stability of the aggregation of rock particles; it is clearly due to the mechanical stability of the matrix and to the mechanical stability of individual particles of rock. Also, it is clear

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[4] Voids due to incomplete filling of mold or form are not considered here, only the voids that are normal components of the mixture.

that the stability of the matrix is due to that of hardened cement paste.

INTERPARTICLE FORCES IN FRESHLY MIXED CONCRETE

We have already seen that plasticity of freshly mixed concrete is possible because the rock particles of the aggregate are slightly separated from one another by matrix material; otherwise, any deformation would necessarily be dilatant rather than plastic. The matrix, itself, is plastic because the cement particles and air bubbles are dispersed in water and especially because the interparticle forces tend to hold particles together while at the same time preventing actual point to point contact. Such a state is due to the coexistence of forces of attraction and repulsion between cement particles. Attraction is due to relatively long range intermolecular forces known as van der Waals forces, about which more later; the forces of repulsion are, in this case, due to electrostatic repulsion and to a "disjoining pressure" (Deryagin)[5] maintained by adsorbed water molecules covering the surfaces of the grains. Electrostatic repulsion is due to what is called a Gouy diffuse layer of ions, in this case negative ions, the negative ions being held near each cement particle by positive ions selectively adsorbed from the surrounding aqueous solution.

Owing to the existence of opposing interparticle forces, a pair of cement grains has a minimum of potential energy with respect to those forces when the particles are separated from each other by a certain small distance, the distance amounting to perhaps ten water molecule diameters, more or less; hence, cement particles tend to assume positions with respect to each other corresponding to minimum potential energy with respect to balance of internal forces, and this is an essential condition for the plastic state.

When particles are in positions of minimum potential energy with respect to the forces acting between them, they are said to be in "potential troughs," and any mechanical displacement of particles with respect to each other requires a certain amount of work to "lift" the particles out of their potential troughs. Thus, we see that interparticle forces give freshly mixed paste in the quiescent state, a structure having a low degree of firmness or shearing strength. When shearing stress exceeding shearing strength is applied and maintained, a paste is caused to flow continuously if its solid content is considerably smaller than that at normal consistency, which is the case for pastes used in concrete. The shearing strength (yield value) and the resistance to continuous shearing stress (the mobility or structural viscosity) is often used as a measure of paste consistency, but such a measure pertains only to paste in the fluid state maintained by a sufficiently high shearing stress.

Consistency of freshly mixed paste can be made softer by diminishing the depth of the potential troughs, and this can be done by using an appropriate surface-active material able to increase interparticle repulsion; indeed, repulsion can be raised to such a degree as to destroy plasticity, changing paste from a plastic to a fluid material. In some concrete mixtures, the water content of the pastes is so high that the paste has very little plasticity to begin with; even though the interparticle forces discussed above exist, the particle concentration is so low that interparticle forces are rela-

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[5] In previous publications I have used terms such as *film pressure, swelling pressure,* or *spreading pressure,* to indicate the force developed by adsorbed films of water in spaces too narrow to accommodate the normal thickness of the films. Several years ago Deryagin, a surface physicist of the Soviet Union, introduced the term *disjoining pressure* and another, *disjoining action;* when thinking of the tendency of water to disperse a coherent system of particles, Deryagin's term seems most apt.

tively ineffective. The paste in a properly constituted freshly mixed concrete has an optimum consistency, neither too soft nor too firm (stiff); under no circumstances should it be completely fluid.

CHEMICAL NATURE OF HYDRATED CEMENT

The chemical compounds found in hydrated cement are complex; most of them are impure in the sense that they contain elements not ordinarily given in their formulas, and they do not have exactly the same composition when formed under different conditions, especially with respect to temperature and original cement concentration. For our present purpose it will suffice to mention a few outstanding characteristics.

All the components of hydrated cement are basic; the hydroxyl-ion concentration is always at least as high as that of a saturated solution of calcium hydroxide, and it is usually considerably higher because of the presence of alkali hydroxides. Any of the compounds can be decomposed by carbonic acid and, therefore, by ordinary rain water. That concrete is not generally destroyed this way is explainable mostly in terms of physical factors: under ordinary conditions of exposure the quantity of acid in contact with concrete during a given time is small relative to the quantities of basic material available, and the permeability of concrete to water is so low that the action of weak acids is only superficial. Even contact with soft-water streams usually causes decomposition at a negligible rate. In cities where rain falls through industry-polluted air and becomes distinctly acid, acid action is evidenced by surface etching. But, whenever a continuous supply of strong acid is encountered, concrete must be protected or it will be destroyed.

Hydrated cement is able to react also with carbon dioxide gas in the presence of water vapor, but the effect is not destructive; actually such reaction may increase chemical stability.

The reactions between the anhydrous components of portland cement and water are remarkable in that they involve a doubling of the volume of space required by solid material while the apparent volume of the system remains constant. There is one exception: the formation of calcium sulfo-aluminate by reactions involving gypsum and tricalcium aluminate tends to cause volume expansion, and when the sulfate ion concentration is too high, expansion can be destructive. Normally, the amount of gypsum needed to control the setting of cement gives only tolerable expansion, but when an unlimited supply of sulfate ion is present in the environment, concrete may be destroyed by it. Practically, such destruction is avoidable by using a cement of low tricalcium aluminate content.

Chemical Aspects of Mineral Aggregates:

The petrographers and mineralogists contributing to this publication have much to say on this subject; I shall mention only some chemical characteristics that are of special interest because of the structural and chemical characteristics of the paste component of concrete.

Rocks used for concrete aggregates are generally materials that have survived geologic ages and are thus those that have demonstrated some degree of chemical and physical stability. Some are chemically basic; some are acidic. Limestone is, of course, basic and vulnerable to acid attack, but when used as concrete aggregate it is less vulnerable than the hydrated cement paste which envelopes it. There are at least two other kinds of undesirable chemical attack on certain minerals that seem to be the result of conditions peculiar to the interior of concrete; one kind involves siliceous rocks; the other, certain dolomites.

Some kinds of siliceous rocks, opal being an outstanding example, may be decomposed by the caustic solutions found in concrete. Under some conditions this reaction is accompanied by destructive expansion, and under other conditions, no expansion. The conditions mentioned involve chemico-physical factors too complex to be described here, as may be ascertained by referring to the literature on the alkali-silica reactions in concrete. A principal factor determining the physical effect of such reactions is the special and selective hindrance to the diffusion of various ions through the structure of cement paste; another is the quantity and specific surface area of the reactive form of silica.

Also, some dolomites react with the caustic aqueous solution in concrete and expand destructively; the reaction involves decomposition of the dolomite and the formation of magnesium hydroxide (brucite); it is called dedolomitization.

Structure of Hardened Cement Paste:

We have already seen that freshly mixed cement paste is a dispersion of cement particles in water and that it has a certain structure owing to the forces of attraction and repulsion among the cement particles. This structure is the starting pattern of the structure that subsequently develops from the materials produced by reactions between the components of cement and water. These reaction products are collectively that which we have already called hydrated cement; now we shall stress the physical aspects of hydrated cement.

Although hardened cement paste looks the way we might expect an amorphous continuum to look, we know that it actually comprises a hierarchy of aggregations of matter. Moreover, we know that cement paste contains submicroscopic voids, its void content usually being upwards of 40 per cent, although a lower void content is possible. Since, in general, we have learned to think of matter as intrinsically granular, we are inclined to regard the pore space as having the character of interstices in a granular aggregation. By dispersing the structure and examining the fragments by electron microscopy, we have seen particles and have been impressed by their smallness and irregularity of shape. These particles, not all of the same kind chemically, may be regarded as the primary particles of paste structure, even though atoms and molecules are the primary and secondary aggregations of matter in general.

The term gel particle refers to particles having dimensions in the submicroscopic range of sizes called colloidal; in this particular case colloidal bodies can be defined as molecular or ionic aggregations having a very high specific surface area, such as is possible only in the submicroscopic range. The colloids observed in cement paste are mostly quasicrystalline, lack of normal crystallinity being due to the extremely small size and imperfect atomic or molecular organization of the solid material. The high specific surface area is due mostly to the thinness of the particles, one of the three dimensions, and perhaps two, being greater than the limit usually stipulated for the colloidal state; these particles are usually only three or four molecules thick.

Along with the colloidal material in hardened cement paste is crystalline calcium hydroxide having relatively low specific surface area. The amount of calcium hydroxide is different for cements having different chemical compositions, but it is usually between 20 and 30 per cent of the weight of the dry hydrated cement. Calcium hydroxide crystals are usually surrounded by and intergrown with colloidal material, and thus they constitute an integral part of the solid structure.

The most abundant colloidal constituent of hydrated cement is an impure calcium silicate hydrate of somewhat indefinite stoichiometry. It has characteristics resembling those of a natural mineral called tobermorite and has thus come to be called *tobermorite gel.* (The term gel designates a rigid aggregation of colloidal material.) There are also amounts of calcium aluminate hydrate and calcium alumino ferrite hydrate; the physical states of these materials are not known exactly, but it appears that they are colloidal but with a lower order of specific surface area than those of tobermorite gel.

An outstanding characteristic of the colloidal matter in hydrated cement paste is that its specific surface area is virtually the same in all pastes made of the same cement regardless of differences in paste density, and among pastes made with different portland cements it is not much influenced by differences in chemical composition. This observation is one of the cornerstones of our concept of paste structure.

The colloidal matter together with calcium hydroxide appear as a continuous solid structure apparently occupying the whole volume of any specimen of hardened cement paste. As already indicated the structure is porous; normally the solid matter occupies 45 to 60 per cent of the apparent volume, and the highest possible solid content (exclusive of unhydrated cement if any) has been found to be about 72 per cent or perhaps a little more of the total volume occupied by hydrated material. In other words, a specimen of hydrated cement paste may have a porosity of not less than about 28 per cent, and it usually has a porosity between 40 and 55 per cent; the porosity will have a higher range if the paste is not fully mature, which means if complete curing has been deliberately or inadvertently omitted. The apparent volume of a specimen of paste, expressed as a volume per unit quantity of cement, is determined by the net volume of mixing water per unit volume of cement. The net volume of mixing water is that which remains within the specimen at the end of the period of settlement (bleeding) which is normally from 1 to 2 hr after mixing.

The solid content of any freshly mixed paste is a little over 62 per cent of what the solid content will be after chemical reactions have converted all the cement to the hydration products described above. As already mentioned, the increase of solid volume takes place without appreciable change of over-all volume,[6] regardless of how high the cement content may be or how little water per unit of cement. One cubic centimeter of cement, solid volume, produces about 1.6 cc of hydrated cement. On this basis we might expect that the hydration products will require 0.6 cc of space in addition to the space originally occupied by 1 cc of cement, or 0.19 cc of space per gram of cement; this amounts to saying that if the water-cement ratio is 0.19 by weight, there will be ample room within the specimen for all hydration products that can be derived from one gram of cement, and the material would become a voidless solid. But experimentally it was found that void space cannot be eliminated. This experimental observation is another cornerstone on which our concept of paste structure rests.

As just indicated, it was found that under no circumstance can completely mature paste be made entirely solid; the highest possible solid content is about 72 per cent, and the rest of the unit volume remains full of water or is void if all the

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[6] The microscopic changes that may occur later as the result of drying and wetting are properly ignored here.

water in such space is caused to evaporate. The densest possible hydrated cement paste contains a continuous system of pores, as evidenced by its permeability to water. Because of the intrinsic porosity of the structure, which limits the solid content to 72 per cent of the apparent volume, it follows that instead of 1.6 cc/cc of cement, the volume of paste must be at least 1.6/0.72 = 2.2 cc/cc of cement to provide enough space for all the hydration products that can be derived from 1 cc of cement, and this means that the water-cement ratio by weight must be at least 0.38. In any paste containing less than this amount of water-filled space, some of the cement remains anhydrous regardless of the duration of curing, and a residue of the original cement remains a permanent feature of the structure of the paste, the residue appearing in cross sections as scattered remnants of the largest grains.

Of course, when the volume of cement paste is greater than 2.2 cc/cc of cement, as it usually is, there is more than enough space to accommodate the hydration products and thus less than 72 per cent of the space can become filled with solid matter. When there is more space available than the minimum required by the hydration products, the extra space is a feature of the structure of paste.

Various observations lead to the concept that in every paste the hydration products tend to become locally concentrated to the maximum degree possible, even when excess space is available, but at the same time they form a continuous structure having an over-all volume equal to the apparent volume of the paste. One line of evidence supporting this view develops from consideration of the structure of freshly mixed paste. Although cement particles in freshly mixed paste are individually dispersed throughout the volume of mixing water, they can not be uniformly spaced because of the interparticle forces, already discussed, that hold the particles practically (but not exactly) in point to point contact. This being true, it seems that if we could subdivide a freshly mixed specimen of paste having a certain water-cement ratio into a large number of cubical cells each having an edge length of say 100 μ (about the same as the mean diameter of the largest cement particles but about 100 times as large as the spherical equivalent of a "particle" of tobermorite gel), we would not find the same volumes of cement and water in each cell; we would find some cells almost filled with a single grain, some would contain many small grains, and some might contain few if any grains. In other words, we would find that the over-all water-cement ratio is an average of many different local water-cement ratios some higher and some lower than the average. Since some of the cells must be nearly full of solid material to start with, considering the size of many of the cement grains, and since interparticle attraction tends to hold particles close together, it seems almost certain that the 72 per cent limit of the content of solid hydrated material will be achieved in many cells after all the cement has become hydrated, even though some of the cells cannot become filled to this extent. In other words, it is reasoned that if the hydrated material in a specimen of paste can reach an average density of 72 per cent but no higher, at any sufficiently low water-cement ratio, that same degree of density can be and is produced locally at various places throughout any paste, however high the water-cement ratio. If any of the imagined cells contain excess cement, which presumably is the case wherever the local water-cement ratio is lower than about 0.38 by weight, such cells can get rid of their excess material by diffusion of material into adjacent cells lacking cement, so that eventually all the cement can become hydrated if the average water-cement ratio is high enough.[7]

I have used the term *cement gel* to designate hydrated cement paste in its densest form. It should be noted that by this definition cement gel is not synonymous with gel as used above, for example, tobermorite gel. The term cement gel is convenient for designating the predominantly colloidal material found in hydrated cement paste, but it must be kept in mind that the term includes noncolloidal calcium hydroxide and other noncolloidal material, if any, and therefore it does not conform exactly to the term gel, properly defined as a solid composed of colloidal material.

The concept of paste structure described above, which requires us to visualize an uneven distribution of cement gel, entails a corresponding uneven distribution of the sizes of interstitial spaces; surface to surface distances range from zero at chemically bonded points to a maximum distance that is probably greater the greater the capillary porosity of the paste, but which in any case is not known exactly. The order of mean pore size is indicated by the quotient of the volume of pore space by the boundary area of that space, which quotient in hydraulic engineering is called the hydraulic radius. For a porosity of 28 per cent and for solid matter having a specific surface area of 5.2×10^6 cm^2/cc of solid matter, which is about the specific surface area of cement gel, the quotient is 7.5×10^{-8} cm or 7.5 A.[8] The corresponding average distance from solid surface to solid surface is between two and four times the hydraulic radius, depending upon the shape of the interstitial spaces; in the present case where many of the spaces are believed to be slit-like, about 18 A seem to be a reasonable estimate. When the porosity exceeds 28 per cent, which is to say when the paste contains spaces other than gel pores, the mean pore size is, of course, greater than the mean size of gel pores.

We can now describe hardened paste as a solid composed of cement gel, remnants of cement grains, if any, and space not filled with cement gel, if any. Any space not filled with cement gel or grain remnants is regarded as interstitial spaces among masses of cement gel and is called capillary space, capillaries, or capillary cavities. The latter term is applied in pastes so dense that the capillaries are discontinuous, the original interstitial space among the cement grains having become segmented into isolated cavities by the growth of cement gel. The pores within cement gel are called gel pores.

Rock Structure:

The structure of concrete is characterized not only by the structure of cement paste but also by the structure of individual pieces of rock making up the aggregate. Some pieces, particularly the smallest, may consist of practically voidless crystals or fragments of crystals; but in most concrete aggregates most of the particles have granular structure and are

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[7] Theoretically, if a 2-in. cube, for example, of fresh paste containing excess cement is joined to one with a deficiency of cement, and if after hardening both are kept saturated with water, the excess cement in one cube could, after the density of hydrated material had reached 72 per cent, diffuse into the excess space in the other; but this conclusion would be hard to prove because the process would probably require geological ages for completion. However, diffusion for a distance of a few microns can occur in a normal curing period, as has been verified by microscopic observations.

[8] To acquire an idea of the magnitude of an angstrom unit, try the following: if one could place two marks one angstrom apart on a piece of superrubber 1 in. long, and then if one could stretch the rubber until it could encompass the earth, the two marks would have become a little over 6 in. apart.

porous, and most are permeable to fluids. The pores in permeable rock are usually larger than those in cement paste; also rocks are less porous than paste, as a rule. A rock having a porosity of say one per cent, may have a coefficient of permeability to water equal to that of a specimen of hardened paste having a porosity of 50 per cent; thus, we know that the pores in rocks are usually larger than those in hydrated cement paste; in other words, rocks have a relatively coarse texture. A few kinds of rock do have fine texture, and unless they are also practically nonporous they do not make satisfactory concrete aggregate; certain argillaceous limestones are examples of unsuitable fine textured rocks; certain cherts also.

Some rocks contain pores that reduce their apparent specific gravity but do not affect their permeability. Among these are vesicular rocks and artificially expanded shales in which isolated voids were formed by expansion of trapped gas. Such rocks are useful when concrete of low unit weight is desired.

States of Water in Concrete:

The solid matter in mature concrete may contain water molecules or ions derived from water, as indicated by the formulas for the compounds in hydrated cement, and by those for some minerals found in concrete aggregate. Of principal interest, however, is the water that remains chemically free, and which is found in gel pores, or in capillary spaces in paste or rock. We shall confine the discussion mostly to chemically free water found in cement paste; it influences the properties of concrete to a very important degree.

It is commonly known that water in a vessel open to the atmosphere will eventually evaporate unless the atmosphere is saturated with water vapor. Cement paste can be regarded as a vessel open to the atmosphere so far as contained water is concerned, but not all of the water it is capable of holding can evaporate unless the surrounding atmosphere is practically empty of water vapor; if the cement paste was originally saturated, some of the water can evaporate, but a definite fraction of it will be retained, the amount retained being a larger fraction of the total evaporable water the higher the degree of saturation of the ambient atmosphere, that is, the higher the humidity. At low ambient humidities, water molecules are restrained from evaporating by the van der Waals forces of attraction between them and the surfaces of the gel particles, which attraction holds a condensed film of water molecules; in other words, the restrained molecules are held by van der Waals forces.

Because of cement paste the internal surface area is very extensive, a large fraction of the total evaporable water can exist as a thin film spread over solid surface. As has already been pointed out, opposite surfaces are necessarily quite close together. In spaces up to about 18 A wide (see above) adsorption of two molecular layers on each surface is sufficient to fill most of such space, and such will be the condition when the ambient humidity is about 50 per cent. At higher ambient humidities, the pore space may be almost completely full, adsorption being aided by hydrostatic tension maintained by curved meniscuses of the water in capillary spaces. A state of mechanical tensile stress between water molecules in the condensed state restrains evaporation in about the same way that van der Waals attraction restrains evaporation from a thin layer on a solid surface.

Of the total capacity of mature cement paste for evaporable water, from about one third to two thirds of it will be full when the ambient humidity is only 50 per cent. In rocks having porosities upwards of one per cent the relatively large pores can retain very little water at that humidity, but at humidities upwards of 90 per cent, a considerable fraction of the total capacity of rock for evaporable water may be retained. Of course, different rocks differ considerably in this respect; the fine textured rocks usually retain a comparatively large amount of water at an intermediate humidity.

The failure of water in concrete to evaporate as it normally does shows that it is altered to some degree by the material with which it is in contact. Some of the alteration may be due to dissolved material, particularly the alkalies, but removal of solutes only modifies the situation. The principal forces acting on water and preventing it from evaporating into an unsaturated atmosphere have already been identified as van der Waals forces of attraction and capillary-induced tension. Naturally, such forces are balanced by counterforces, the counterforces corresponding to elastic strains of one kind or another in the solid structure within which the water is held. Such strains correspond to the reversible part of volume changes due to drying and wetting of concrete.

There is good evidence that some of the evaporable water normally held in concrete contributes to its strength, that is, van der Waals attraction between water molecules and between solid material and water molecules seems to contribute to the total cohesive force. However, there is a somewhat greater weakening effect due to swelling.

Strength:

In the above discussion dealing with cohesion and adhesion we necessarily spoke of strength. But there are other aspects of the strength of concrete depending on the nature of its structure, and some of these will be discussed in the following paragraphs.

It would be expected that with a given aggregate the more cement gel per unit volume of paste and the more paste per unit volume of matrix, which means the lower the air content of the matrix, the greater the strength of the concrete. Strength tests of many kinds of concrete show that such is indeed the case; it was the fact underlying the cement-space ratio law for strength given by Feret (1897), and the less general water-cement ratio law given by Abrams (1918), and earlier by Zielinski (1908). For aggregates composed of strong particles, the upper limit of concrete strength tends to be established by the upper limit of the density of the matrix, that limit usually being established by the means at hand for compacting the mixture. On the other hand, with aggregates composed of relatively weak particles, the upper limit tends to be established by the strengths of the aggregate particles. This does not mean, however, that concrete cannot be stronger than the strength of individual aggregate particles, as is evident when one considers air bubbles as aggregate.

The strength of a given kind of concrete is not a single valued property of the material. Compressive strength, in particular, is a function of the rate at which stress is applied, the function being such that strength appears higher the higher the rate of loading. If a stress less than the strength indicated by an ordinary test procedure, and yet greater than about two thirds of that stress, is maintained without increase, the specimen will eventually fail. There are good reasons to believe that failure is essentially a random (stochastic) process, and therefore the stress existing at time of failure is intrinsically a variable number; in other words, two absolutely identical specimens would not be likely to fail at exactly the same stress, or, under a sustained high stress less than the mean "instantaneous" strength, they are not likely to fail at the same elapsed time after loading. Thus, it seems that even with a perfect testing machine and with test specimens exactly alike, we would still find variation of test results about the mean value from a large number of identical specimens.

When a concrete cylinder is subjected to a steadily increasing axial stress, as in the ordinary compression test, the observed increase in diameter at low stress is a certain constant fraction (Poisson's ratio) of the longitudinal shortening (compressive strain). There is no tensile stress associated with the lateral strain just mentioned, but after the compressive stress becomes about two thirds of the stress at failure, further increase in compressive stress causes lateral dilation to increase more than can be accounted for by Poisson's ratio, and this extra strain does denote the development of tensile stress. When such stress begins to exceed tensile strength, vibrations due to internal splitting can be detected with suitable instruments. At failure, fractures appear as uneven surfaces having an inclination to the axis somewhat less than 45 deg, or fractures appear as cracks parallel to the axis, or both kinds of fractures appear. Such experimental evidence indicates that the failure of concrete under compression, or when subjected to shear stress, is essentially failure in tension. If failure under compressive stress were limited only by strength in pure shear, the principal strains preceding failure would involve only the sliding of one smooth surface over another, and thus no increase in volume other than that accounted for by Poisson's ratio would be necessary; hence, the observed extra dilation with simultaneous internal cracking shows that tensile stress develops across the incipient fracture surfaces and causes separations at such surfaces.

Current theory of fracture indicates that during a compression test tensile stress necessarily develops around holes, cracks, or flaws in the material, and thus tensile stress would develop even in a continuum, structureless except for flaws. Without questioning this deduction we can at the same time suggest that tensile stress would arise anyway, simply because of the granular nature of the material. Since the individual particles of which paste or rock are composed are much stronger than the structure, it is impossible for the surface of a fracture in paste to be smooth. Specimens of neat cement show evidence of tension failure in a compression test; they have a strong tendency to split. In concrete made of strong aggregate material and weak paste, the nominal shearing surfaces are likely to follow the contours of rock particles, in which case the cause of tensile stress is obvious; in concrete made with strong pastes, fracture surfaces usually pass through rock particles, but even so the fracture surface is not smooth, and again it is not difficult to account for the development of tensile stress as one rough surface is forced to move away from the other as it tends to slide. These considerations amount to classifying cement paste or concrete as an intrinsically dilatant system, just as compact, uncemented, granular systems are intrinsically dilatant.

CONCLUSION

While discussing the structure of concrete and the internal forces that give it stability, various properties were discussed. It would be possible to elaborate these brief discussions and to add other topics; however, other papers in this publication are concerned with such reviews, and no doubt interpretations in terms of structure will be found in them. Therefore, this paper is concluded without further development of the subject.

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Freshly Mixed Concrete

UNIFORMITY AND WORKABILITY

By C. A. Vollick[1]

Synopsis

Deviations in materials, practices, and conditions that influence within batch or batch to batch variability of concrete properties and tests used to measure these deviations are discussed. Methods used to control concrete variables within acceptable limits are described. The significance of workability and tests currently used to measure this elusive property of concrete are reviewed.

Concrete is a manufactured product. Any successful manufacturing operation requires that the products produced from day to day are uniform and similar in appearance and quality. Concrete manufacturing differs from other types of manufacturing in many important respects which enhance the versatility of concrete for building construction, but increase the problems of quality control and uniformity.

Uniformity of Concrete

Concrete is a heterogenous mixture of natural or artificial aggregates, cement, water and occasionally entrained air, pozzolans, and other admixtures. The materials used in the manufacture of concrete have wide ranges in physical and chemical composition, and these variations are reflected in the workability, appearance and quality of the concrete.

Nonuniformity may be evident in freshly mixed or hardened concrete. There are two distinct classes of nonuniformity in freshly mixed concrete, that is, within-batch variations and batch-to-batch variations.

Concrete initially discharged may vary in many important respects from the last concrete in the mixer, or variations may occur throughout the batch. These differences, which are classed as within-batch variations, may be attributed to blade wear on the mixer, inadequate mixing, improper loading sequence, or overloading of the mixer.

Differences in appearance, workability, and quality of concrete placed on different days or from different mixers on the same day are designated batch-to-batch variations. These variations may occur as the result of changes in one or more of the following:

1. Mix proportions of aggregate, cement, water, and admixtures, including batching errors.
2. Moisture content of aggregates.
3. Aggregate grading.
4. Type or brand of cement.
5. Different lots of cement from the same source.
6. Type and shape of aggregates.

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7. Concrete temperature.
8. Air temperature.
9. Type of mixing equipment.
10. Testing procedure.

After mixing, every operation involved in the transportation, depositing, and consolidation of concrete in the forms can contribute to segregation and nonuniformity. Inadequate preparation of forms may also influence nonuniformity by permitting sand, cement, and water to escape, with a resulting accumulation of coarse aggregate, in certain areas.

The properties of the concrete in the finished structure are of primary importance. Properties and tests of the individual components and of the fresh concrete are valuable aids in predicting and controlling the uniformity, homogeneity, and quality of the finished product. Experience has shown that variations affecting the properties of plastic concrete will also affect the properties of hardened concrete.

CONTROL OF CONCRETE PRODUCTION

Production of uniform concrete can be obtained only through systematic control of all operations from selection and production of materials through batching, mixing, placing, consolidation, and curing of the concrete. The materials supplied batch after batch to the mixer must have uniform properties. Control must be maintained at the batching and mixing plants. Aggregates should be tested for gradation and moisture content, and mix adjustments made to correct for changes in these properties. Mixed concrete should be tested for consistency, air content, temperature, and unit weight. Concrete cylinders should be fabricated for compressive strength tests.

Routine instructions for measuring, mixing, and placing concrete are given in the American Concrete Inst. (ACI) Manual of Concrete Inspection [*1*].[2] Additional information on practices that lead to better uniformity are found in the recommendations of ACI Committee 614 [*2*]. Some variation must be accepted, but consistent concrete of satisfactory quality can be obtained if proper control is maintained.

Concrete uniformity has generally been measured in terms of compressive strength, slump, unit weight, air content, and content of coarse aggregate or cement. Uniformity tests have been used to establish required mixing time, mixing speed, mixer capacity, and efficient batching procedures. Tests by a number of investigators have been considered in the preparation of ASTM Specifications for Ready-Mixed Concrete (C 94).

METHODS OF MEASURING UNIFORMITY

Dunagan Test:

Dunagan [*3*] proposed a method of measuring the proportions of cement, water, sand, and coarse aggregate in fresh concrete by a series of wash separations and weighing in air and water. This method has been used by Slater [*4*], Hollister [*5*], Cook [*6*], and others to study the effects of different rates of rotation of truck mixers, effect of time of haul, and effect of mixing time on uniformity of concrete. The Dunagan test has limited usefulness because of sampling errors and difficulties in distinguishing between cement and very fine sand.

Air-Free Unit Weight of Mortar:

The U.S. Bureau of Reclamation evaluates the performance of stationary mixers by comparing the air-free unit weights of mortars [*7*]. Two samples of concrete are obtained from different

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

locations in a mixer. Volumes of the samples are determined, and they are weighed, washed over a No. 4 screen, and the saturated surface-dry weight of the coarse aggregate determined. The air-free unit weight of the mortar is calculated by formula which includes specific gravity of the coarse aggregate. Large variations in this test may indicate that batching procedure is inefficient, or mixer blades are worn. Additional mixing time may be required if the unit weight varies more than 1.5 lb/ft^3.

Bureau data for one series of mixing time tests on 4 yard^3 mixers showed that variation in air-free unit weight was reduced from 3.0 lb to approximately 1.1 lb when the mixing time was increased from 1 to 2 min, but increased mixing beyond 2 min had little additional influence on this variable. Variation in water-cement ratio was reduced from 29 to 5 per cent when mixing time was increased from 1 to 2 min in this same series of tests.

A study designed to establish test methods and limits for variations in truck mixed concrete was reported by Bloem, Gaynor, and Wilson [*8*]. Variations in slump, air content, per cent of coarse aggregate, air-free unit weight of mortar, water content by oven drying, and compressive strength of concrete obtained after approximately $\frac{1}{6}$, $\frac{1}{2}$, and $\frac{5}{6}$ of discharge from a truck mixer were determined. They concluded that the air-free unit weight test was an improvement over the unit weight test because the number of variables was reduced and excessive changes in this property reflected changes in water or in proportions of cement and sand. According to their data, a difference in air-free unit weight of mortar of 1 lb/ft^3 corresponded to a change in water of about 2 gal/yard^3 when the proportions of sand to cement were maintained constant and the water alone was varied. They suggested that a variation of more than 1 lb/ft^3 in this test indicates real differences in the proportions of the mortar ingredients, and differences of more than 2 lb/ft^3 should be considered evidence of unsatisfactory uniformity.

Centrifuge Test:

This test, also known as the Willis-Hime Method, is described in detail in *ASTM Bulletin*, No. 239 [*9*]. It provides a basis for within-batch comparisons of cement content of concrete and employs a liquid with a density greater than sand but less than cement. This liquid is used to separate the components of a carefully prepared mortar sample extracted from the concrete. This test is one of several mixer performance tests specified in ASTM Specifications for Ready-Mixed Concrete (C 94). Variability of seven per cent or more in the cement content of two samples from the mixer is evidence of incomplete mixing.

The centrifuge test was used in the study by Bloem, Gaynor, and Wilson [*8*]. They concluded that the test is quite involved, and in most cases the information gained is not commensurate with the time and labor required. Data cited from previous tests indicated that the test was highly reproducible provided extreme care was used and corrections were made for sampling errors in coarse aggregate content of the small test portions.

Unit Weight:

Unit weight of the concrete in influenced by the specific gravity and amount of coarse aggregate, air content, proportions of sand to cement, and water content. Consequently, variations in unit weight are difficult to evaluate as to cause or significance. The test is more definitive when the weight and solid volume of coarse aggregate and volume of air are eliminated as in the air-free unit weight of mortar test.

The unit weight test is recommended as a job control measure for lightweight aggregate concrete in conjunction with air determinations and slump. If the slump and air content are maintained constant, a change in unit weight indicates a change in weight of aggregate. If the weight of aggregate per cubic foot of lightweight concrete changes, it may be the result of change in moisture content, gradation, or density of the aggregate. Additional tests, including unit weight, moisture content, and gradation of the aggregate are required in order to determine the cause of the change.

Air Content:

Air content has an important influence on workability, compressive strength, and durability of concrete. The strength of concrete decreases uniformly with increase in air content of the fresh concrete provided the water-cement ratio is held constant. Air entrainment also increases slump, each one per cent additional air being approximately equivalent to 1 in. in slump. When the slump is maintained constant by reduction in water content, strength reduction due to air-entrainment is not so great and the strength of lean mixes may be slightly increased. Strength reductions of 16 to 20 per cent at 28 days have been reported for concretes containing 5.5 to 6.5 sacks of cement per cubic yard when five per cent entrained air was added to the concrete.

Too little air will not provide workability and durability. Sudden loss of workability may indicate a reduction in air content. Resistance of concrete to freezing and thawing is increased several hundred per cent by incorporating the correct amount of air in the concrete. Consequently, it is important that the concrete contain a uniform quantity of air. Within-batch variations should not exceed one per cent.

Variation in air content obtained from a given dosage of air-entraining agent may result from changes in concentration of the agent, brand or type of cement, sand grading, pozzolan, or admixture, temperature of the mix, slump, or length of mixing. Air content is generally maintained at the correct level by increasing or decreasing the dosage of air-entraining agent because it may not be practical or desirable to remove the cause of the variation. When air-entraining cement is used, control of air content is more difficult because any change in the dosage of air-entraining agent must be accompanied by a corresponding change in cement content. For this reason many engineers prefer to use regular cement and add the air-entraining agent at the mixer.

Air content of normal weight concrete may be computed by comparing the actual unit weight of concrete with the theoretical unit weight based on the specific gravity of the materials used, as outlined in ASTM Test for Weight Per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138). Results obtained by this method are influenced by variations in mix proportions, specific gravity of ingredients, and changes in moisture content of aggregates. Furthermore, any change in batch weights requires a new computation.

Several meters have been developed to determine the air content of fresh concrete directly. Determination of air content by this method is generally preferred to the gravimetric or unit weight method. The American Society for Testing and Materials has standardized two methods of determining air content by means of meters.

The pressure meter (ASTM Test for

Air Content of Freshly Mixed Concrete by the Pressure Method (C 231)) consists of a special pressure-tight container and accessories designed to hold an accurate volume of concrete. The container is filled with concrete, the lid is securely fastened, and pressure is applied by hand pump to a predetermined amount. The apparatus is so calibrated that the percentage of air is read directly when the pressure is released. This method is used more than any other and is considered satisfactory for all types of concrete and mortar except those made with highly porous lightweight aggregate. This apparatus must be calibrated periodically to guard against changes caused by rough usage and, if the elevation of the place at which the apparatus is used changes by more than 600 ft, it should be recalibrated. An aggregate correction factor should be determined with the materials used and subtracted from the apparent reading to determine the actual air content. The aggregate correction factor varies only slightly for the same type of aggregate and need only be checked when there is a definite change in materials.

The volumetric method (ASTM Test for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173)) consists of removing air from a sample of concrete by mixing it with water in a special container. The volume of air is determined from the difference in volume of the sample containing entrained air and the volume of the sample after it has been agitated to permit the air to escape. This method is recommended particularly for lightweight concrete, but it may be applied to other types of concrete as well.

The air indicator is a miniature device that uses the volumetric principle. A small sample of carefully selected mortar is obtained from the concrete and placed in a brass cup measuring $\frac{3}{4}$ in. in diameter by $\frac{1}{2}$ in. high and compacted with a wire or knife blade. The glass tube that comes with the device is filled with isopropyl alcohol to the top line, the brass tube is inserted in the tube, and the liquid level is adjusted to the top line. The finger is placed over the stem to prevent alcohol from escaping, and the indicator is rolled several times until all mortar has been removed from the cup. With the indicator in a vertical position the finger is carefully removed from the stem, and the number of graduations from the top to the new liquid level gives an indication of the air content in the mortar sample. A correction factor, based upon the design mix, must be applied to convert per cent air in concrete. Meticulous care must be used in selection of the mortar sample, method of inserting the stopper, agitation of the sample, and removal of the finger from the tube. The test can only provide an indication of relative air content and cannot be considered as reliable as the pressure meter or the volumetric method.

Slump:

The slump test (ASTM Test for Slump of Portland Cement Concrete (C 143)) is essentially an indication of the wetness of the concrete. Within-batch variations in slump indicate incomplete mixing and nonuniform distribution of water or other ingredients throughout the batch. Batch-to-batch variations may result from batching errors, uncorrected changes in moisture content or grading of the aggregate, or variations in temperature. In reasonably uniform concrete, slump should not vary more than about 1 in. within a batch.

Compressive Strength:

Measured concrete strength is used widely as a criterion of concrete quality. Other factors such as durability may be more critical, but strength tests are easily made and variations in strength indicate variations in other properties.

Compressive strength is a control test used to determine the degree of uniformity of concrete. ACI Committee 214 [*10*] has suggested statistical methods useful in interpreting variations in concrete strength. ASTM Committee C-9 has suggested as a tentative revision to Designation C 94 that within-batch variation in compressive strength of ready-mixed concrete should not exceed 7½ per cent at seven days. In a well controlled laboratory, compressive strength of cylinders fabricated from the same concrete sample may vary three to five per cent. Variations in excess of this amount must be attributed to variations within the batch of concrete.

WORKABILITY

Workability is a term that is used every day in concrete construction, and it is a factor easily appreciated in practice. Workability means different things to different people and for different placing conditions. Various methods have been developed for its measurement. None of these tests evaluate all characteristics that are involved in this property.

Glanville [*11*] defined workability as: "that property of the concrete which determines the amount of useful internal work necessary to produce full compaction." Powers [*12*] defined it as: "that property of the plastic concrete mixture which determines the ease with which it can be placed and the degree to which it resists segregation." Both of these definitions are expressed in terms related to the physical characteristics of the concrete alone, being independent of the methods of placing and compacting.

Workability of concrete is defined in ASTM Terms Relating to Concrete and Concrete Aggregates (C 125) as: "that property determining the effort required to manipulate a freshly mixed quantity of concrete with minimum loss of homogeneity."

In actual practice, workability is related directly to the type of construction and methods of placing, mixing, and working. Concrete that can be placed readily without segregation in a mass dam would be entirely unworkable in a thin structural member. Workable concrete compacted by means of high-frequency vibrators would be unworkable if vibrators could not be used and hand tamping and spading were required. Concrete having suitable workability for a pavement might be unsuitable for use in a thin, heavily reinforced section.

Properties involved in workability include finishing characteristics, consistency or fluidity, pumpability, mobility, segregation, and bleeding. None of the test methods proposed or in use today simultaneously measure all of these properties. Consequently, measurement of workability is determined to a large extent by judgement, based on experience.

Workability is dependent upon the physical and chemical properties of the individual components and the proportions of each in the concrete. The degree of workability required for proper placement and consolidation of concrete is governed by the type of mixing equipment, size, and type of placing equipment, method of compaction, and type of concrete.

FACTORS AFFECTING WORKABILITY

Some of the factors that affect the workability [*13*] of concrete are quantity of cement, characteristics of cement, consistency, grading of fine aggregate, shape of sand grains, grading and shape of coarse aggregate, proportion of fine to coarse aggregate, percentage of air entrained, type and quantity of pozzolan, quantity of water, and amount and characteristics of admixture used.

Cement:

Very lean mixes tend to produce harsh concrete having poor workability. Rich mixes are more workable than lean mixes, but concrete containing a very high proportion of cement may be sticky and difficult to finish. An increase in the fineness of cement increases the cohesiveness of the concrete mix as well as the rate at which cement hydrates and the early strength development. Differences in bleeding tendency, accumulation of laitance, and other properties of concrete made with cements having the same fineness and chemical analysis have been observed.

Consistency:

The terms consistency and plasticity are terms often used to indicate workability. Consistency generally denotes the wetness of the concrete which is commonly measured by the slump test. It must not be assumed that the wetter the mix the more workable the concrete. If a mix is too wet, segregation may occur with resulting honeycomb or sand streaking on the exposed surface; finishing properties will be impaired because of the accumulation of laitance on the surface. If a mix is too dry it may be difficult to place and compact, and segregation may occur because of the tendency for larger particles to roll towards the outer edge of the heap, formed when it is deposited. It is agreed generally that concrete should have the driest consistency that is practicable for placement with proper vibration. The consistency necessary for full compaction varies with the type of structure, type and size aggregate, and type of compaction equipment available.

Sand:

Concrete containing fine sand requires more water for the same consistency, as measured by the slump test, than an equivalent amount of coarse sand. Very coarse sand can have an undesirable effect on finishing quality. Neither very fine or very coarse sand is desirable. Rounded river sand gives greater workability than crushed sand composed of sharply angular pieces with rough surfaces. Angular sand particles have an interlocking effect and less freedom of movement in the freshly mixed concrete than smooth rounded particles. Natural sand may give satisfactory results with a coarser grading than would be permitted with crushed sand. In addition, concrete must usually contain two to three per cent more sand by absolute volume of total aggregate and 10 to 15 lb more water per cubic yard when crushed sand is used.

Coarse Aggregate:

The particle size distribution of coarse aggregate influences water requirements and workability of concrete. Coarse aggregates meeting standard grading requirements such as ASTM Specifications for Concrete Aggregates (C 33) should be used. After the grading is established, it should be maintained within rather close tolerances to avoid sudden changes in workability and other concrete properties. Segregation is reduced and uniformity improved by separating the aggregate into several size fractions and recombining these fractions when concrete is manufactured.

Breakage, segregation, and contamination of aggregate can occur during handling and stockpiling. Introduction into the mixer of a large quantity of undersize material which may have accumulated will result in a sudden change in workability resulting in a demand for additional water. ACI Committee 614 [*2*] has suggested screening at the batch plant as the method most likely to eliminate undesirable undersize and promote uniformity.

Production of workable concrete with

sharp, angular, crushed aggregates generally requires more sand than similar concrete made with rounded aggregates. The water content may be increased 15 to 25 lb/yard^3. If the water-cement ratio is held constant, more cement is required. Flat or elongated particles which are defined as particles having a ratio of width to thickness or length to width respectively, greater than three are detrimental to concrete workability. More sand, cement, and water are required when the coarse aggregate contains flat and elongated particles.

The maximum size of aggregate that can be used to produce workable concrete is limited by practical considerations including type and size of structure, amount and spacing of reinforcing bars, method of placing, and availability of materials. Generally, aggregate should not be larger than three fourths of the maximum clear spacing between reinforcing bars nor larger than one fifth of the wall thickness or narrowest dimension between sides of forms. ACI 613 [*14*] provides recommendations on the maximum sizes of aggregate for various types of construction. Bureau of Reclamation [*15*] experience in pumping concrete indicates that concrete containing $2\frac{1}{2}$-in. max size aggregate can readily be pumped through an 8-in. pipe, but aggregate larger than $2\frac{1}{2}$ in. may cause difficulty. Concrete containing aggregate graded to 1-in. max size can be placed by pneumatic equipment.

Air Entrainment:

Entrained air increases the paste volume, acts as a lubricant, and improves the workability of concrete. It reduces bleeding and segregation during handling and placing of concrete and increases cohesiveness or "fattiness" of the concrete. Improvement in workability resulting from air entrainment is more pronounced in lean mixes that are harsh and unworkable because of poor aggregate grading, or type of aggregate used.

Finely Divided Materials:

Addition of finely divided material, including inert or cementitious materials or pozzolans, generally improves the workability of the concrete. Improvement is more noticeable in lean mixes than in rich mixes. These materials have been used to improve the grading of sands deficient in fines. Cementitious and pozzolanic materials are usually substituted for 10 to 25 per cent of the cement. Workability will be improved if these materials are added as replacement for part of the sand, instead of substituted for part of the cement.

Chemical Admixtures:

Water-reducing admixtures, when added to concrete, permit a reduction in mixing water with no loss in slump, or, if the water content is held constant, produce an increase in slump. Set retarding admixtures reduce the early rate of hardening and permit concrete to be handled and vibrated a longer period of time.

It has been reported that there is a decrease in the frequency of plugged pump lines when water-reducing retarders are used in the concrete. In addition, less power may be required to pump the concrete.

Mix Proportions:

Workability can be controlled by proper proportioning of the constituent materials. As the proportion of mortar, including sand, cement, water, and air, is increased, the grading and angularity of the coarse aggregate become less important. There should be sufficient mortar to fill the voids in the coarse aggregate plus a sufficient amount to permit the concrete to be placed readily in forms and vibrated around reinforcement. An excess of mortar increases workability, but excess workability is inefficient. It should not be more than is required for consolidation by modern equipment. The quantity of mortar required to produce the desired workability with a given coarse aggregate can be determined most effectively by laboratory tests. ACI Recommended Practice for Selecting Proportions for Concrete provides a basis for estimating the proportions of coarse aggregate to be used in trial mixes.

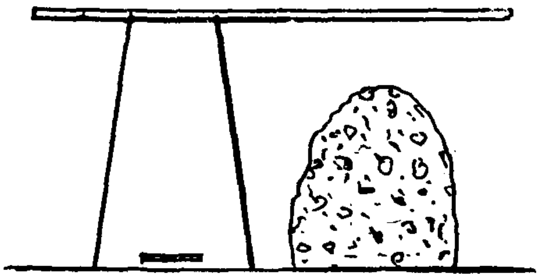

FIG. 1—Slump test.

METHODS OF MEASURING WORKABILITY

Slump Test:

The slump test (see Fig. 1) is the most commonly used method of measuring consistency or wetness of concrete. It is not suitable for very wet or very dry concrete. It does not measure all factors contributing to workability, nor is it always representative of the placeability of the concrete. However, it is used conveniently as a control test and gives an indication of the uniformity of concrete from batch-to-batch. Repeated batches of the same mix, brought to the same slump, will have the same water content and water-cement ratio provided weights of aggregate, cement, and admixtures are uniform and aggregate grading is within acceptable limits. Additional information on workability of the concrete can be obtained if, after removing the slump cone, the concrete is tapped on the side with the tamping rod. Two concretes with the same slump may behave differently, that is, one may fall apart after tapping and be harsh with a minimum of fines, and the other may be very cohesive with surplus workability. The first concrete may have sufficient workability for placement in pavements or mass concrete, but the other concrete may be required for more difficult placement conditions.

The slump test should be performed in strict accordance with the requirements of ASTM Designation C 143. Tests are usually made at the point of placement and should be made whenever cylinders are molded for compressive strength testing. Slump tests may be made at the mixing plant in order to check the uniformity of batching operations.

Popovics [*16*] has presented data indicating that the relationship between consistency values, as measured by the slump test and the water content of concrete, is parabolic, that is, the percentage change in water content required to increase the slump 1 in. may vary from 2.0 per cent when the initial slump is 5 in. to approximately 4.5 per cent when the initial slump is 2 in. An average change in water content of 3 per cent generally is considered necessary for a 1-in. change in slump.

As the temperature of the concrete increases, the slump decreases. Concrete placed at a slump of 4 in. at 70 F may only have 3-in. slump when placed at 90 F, or the same concrete may have a slump of 5.5 in. when placed at 50 F.

Air entrainment and water-reducing admixtures will increase slump of concrete if all other conditions remain the same. Each one per cent increase or decrease in air content will produce approximately the same influence as a change in water content of three per cent.

The slump generally is reported to the nearest ¼-in. Slumps reported by different operators on the same batch of concrete may vary by as much as ½-in. The most unsatisfactory form of slump is the shear slump, that is, a falling away or shearing off of a portion of the concrete from the mass. It is impossible to lay down rules on the correct point to measure the slump. If this condition exists, the concrete probably lacks the necessary plasticity for the slump test. It may be difficult to consolidate in the forms, but if consolidation of the concrete is practical, the slump test is not a valid control test for this type of concrete.

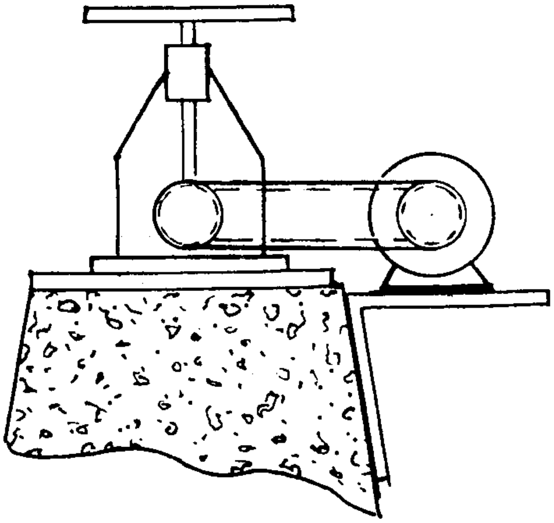

FIG. 2—Flow table.

Flow Test:

The flow test (see Fig. 2) (ASTM Test for Flow of Portland Cement Concrete by Use of the Flow Table (C 124)) has been used primarily in the laboratory to measure consistency of concrete. The concrete to be tested is cast in a metal mold in the shape of the frustrum of a cone, having a base diameter of 10 in., top diameter of 6¾ in., and height of 5 in., on top of a metal table. After the concrete is cast and rodded the mold is removed. The table is then raised and dropped ½ in. with a jolt 15 times in approximately 15 sec by turning the cam at the rate of 1 rps. The increase in diameter of the concrete expressed as a percentage of the original diameter of 10 in. is used as the measure of the flow of the concrete.

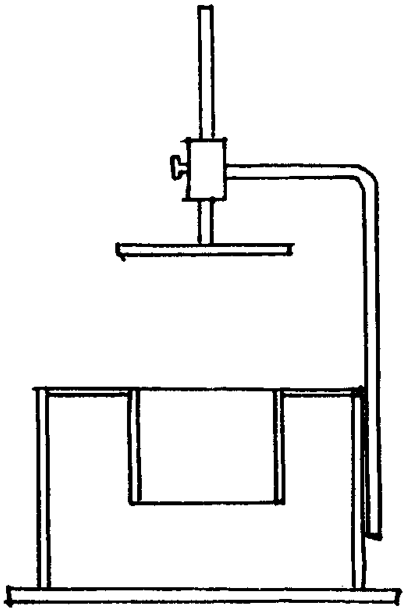

FIG. 3—Remolding apparatus.

The flow table will record consistencies over a similar range to the slump cone. It has not been used widely for field control because the slump test is more convenient.

Remolding Test:

The Remolding Test apparatus (see Fig. 3) was developed by Powers [*12*] to measure "the relative effort required to change a mass of concrete from one definite shape to another by means of jigging." The equipment consists of a metal cylinder mounted inside a larger cylinder and a suspended plate which fits inside the smaller cylinder. A slump cone is placed inside the smaller cylinder so that the bottom rests on the base. It is filled with concrete, the slump cone removed, and the plate rested on top of the concrete. The flow table on which the apparatus is mounted is then operated. The number of ¼-in. drops, required to mold the concrete to a cylindrical form, is a measure of the workability of the concrete. This method has not found widespread use and has not been made an ASTM standard.

The remolding test and slump test were used by W. A. Cordon [*17*] in an extensive series of tests on air-entrained concrete. He found that workability, as measured by the remolding test, was increased when air content was increased, and slump and per cent sand were maintained constant. When per cent sand was reduced as the air content was increased and slump was constant at 4 in., the remolding effort was also constant at approximately 42 jigs. It might be concluded that the remolding test is more sensitive to changes in air content than the slump test.

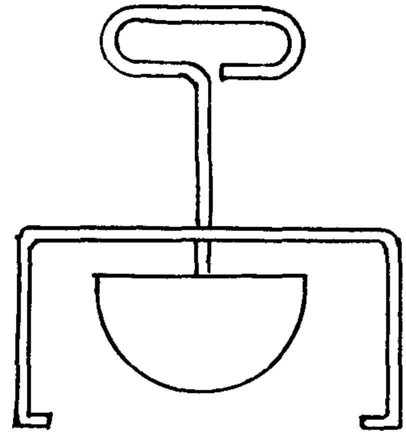

FIG. 4—Ball penetration apparatus.

Ball Penetration Test:

The Kelly ball test (see Fig. 4) [*18*] was developed principally as a convenient method of measuring and controlling consistency in the field. The ball test can be performed on concrete in the forms, and it is claimed that tests can be performed faster and precision is greater than with the slump test. One disadvantage of this test is that it requires a large sample of concrete.

The apparatus weighs 30 lb and consists of a 6-in. diameter ball and stem which can slide through the center of a stirrup, the legs of which rest on the concrete to be tested. The depth of concrete must be at least 8 in., and the minimum distance from the center of the ball to the nearest edge of the concrete is 9 in.

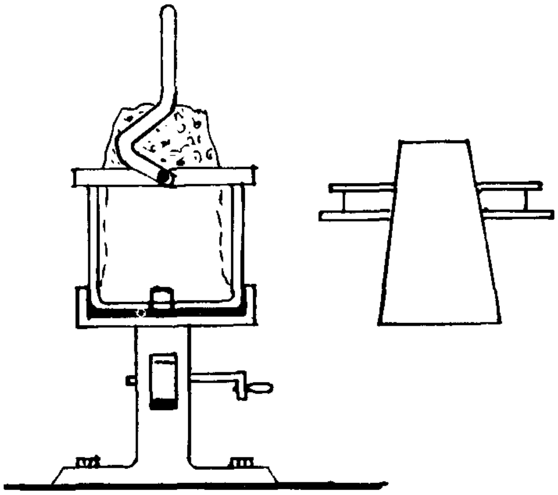

FIG. 5—Thaulow concrete tester.

The surface of the concrete is struck off level, avoiding excess working. The ball is lowered gradually onto the surface of the concrete, released, and the depth of penetration read immediately on the stem to the nearest ¼ in. The ratio of slump to the penetration of the ball is between 1.5 and 2 and is fairly constant for a given mix but varies according to the mix. The method has been adopted by ASTM (Test for Ball Penetration in Fresh Portland Cement Concrete (C 360)).

The ball penetration test was compared with the slump test by Howard and Leavitt [*19*]. Twenty tests were made using each method. The ball penetration averaged 1.32 with a standard deviation of 0.45 and the slump averaged 2.5 with a standard deviation of 0.81. They concluded that the slump test required approximately 10 min, and the ball penetration only required 10 sec when used on paving concrete.

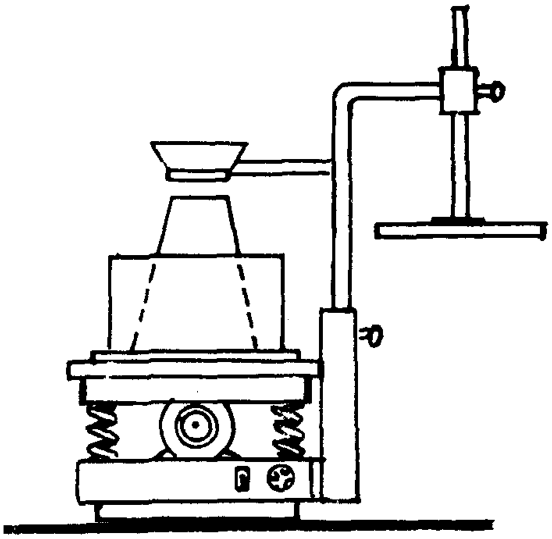

FIG. 6—Vebe apparatus.

Thaulow Concrete Tester:

The Thaulow concrete tester (see Fig. 5) [*20*] consists of a 10 liter container with a gradation mark at 5 liters, equipped with a slump cone, and fitted with a stainless steel handle, and a drop table actuated by a crank which drops the table through 0.394 in. four times for each revolution.

The slump cone is fastened in the container, filled with concrete in the usual manner, and lifted off. The handle is allowed to fall freely from the vertical position alternately on each side of the container until the concrete is remolded and the entire periphery is at the 5 liter mark. The number of blows required is an indication of the workability.

If the concrete is very dry, the container is fastened to the drop table. The slump cone is fastened, filled in the usual manner, and further compacted by 15 drops of the table. The cone is removed, and the number of revolutions of the crank handle necessary to bring the concrete to the 5 liter mark is a measure of the consistency.

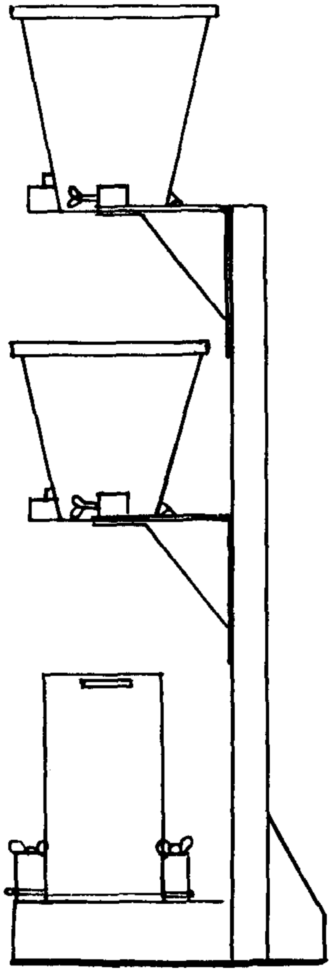

FIG. 7—Compacting factor apparatus.

ACI Committee 211 has prepared a Recommended Practice for Selecting Proportions for No-Slump Concrete. The concrete has a slump less than 0 in. Differences in consistency of very dry mixes cannot be measured with the slump cone, but the Thaulow equipment is considered to have merit for this application. Concrete with a slump of 1 to 2 in. requires 14 to 28 drops, and concrete with a slump of 3 to 4 in. requires less than 7 drops.

Vebe Apparatus:

The Vebe consistometer (see Fig. 6) [*21*] consists of a vibrating table, a sheet metal pan, slump cone, and plastic plate attached to a graduated, free-moving rod which serves as a reference end point. The cone is placed in the pan, filled with concrete, and removed. The plastic disk is brought into position on top of the concrete, and the vibrating table set in motion. The number of seconds required to remold the cone of truncated concrete to the shape of the cylinder is the measure of consistency and is reported as the number of Vebe seconds or degrees. This method is very suitable for very dry concrete, but the vibration is too vigorous for concrete with a slump greater than about 2 in. For example, 0 to 3 Vebe sec is required for concrete with a slump of 3 to 4 in., and 10 to 52 Vebe sec may be required for concrete with less than 0-in. slump.

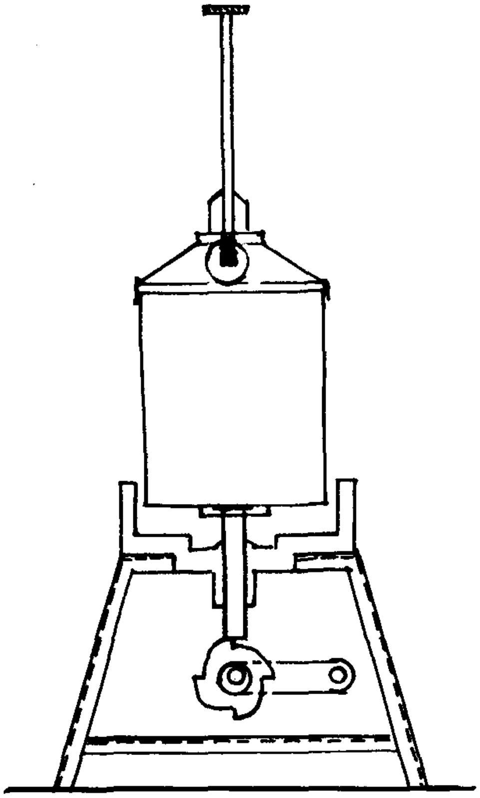
FIG. 8—Wigmore consistometer.

Compacting Factor:

The compacting factor test (see Fig. 7) has been developed in Great Britain [*11*]. The apparatus consists of two conical hoppers fitted with strong doors at their base and a 6 by 12-in. cylinder. The top hopper is filled with the concrete to be tested and struck off without compacting it. The door at the bottom of the hopper is opened, and the concrete drops by gravity into the somewhat smaller hopper below. The door of the second hopper is opened, and the concrete is allowed to fall into the cylinder which is struck off and weighed. The ratio of the weight of concrete in the cylinder mold to the weight of concrete from the same batch fully compacted in the mold is the compacting factor. The sensitivity of this test is considered good for medium consistency concrete, but less than some other tests for very dry concrete. An average compacting factor of 0.75 corresponds to a slump of 0 to 1 in., 0.85 is approximately 1 to 2-in. slump, and 0.90 is approximately 3 to 4-in. slump.

Wigmore Consistometer:

The Wigmore consistometer (see Fig. 8) is described by Orchard [*24*].

This apparatus consists of a galvanized container and hand-operated compaction table. A sliding stem to which is fastened a 2-in. diameter ball is mounted in the lid of the container. The container is filled with concrete which is compacted on the table by 8 drops. The container is again filled with concrete, levelled off, and the lid and the ball are placed in position with the ball resting on the surface of the concrete. The apparatus is placed on the table and the concrete compacted by turning the handle attached to the cam at the rate of about 1 rps. The table drops $\frac{7}{32}$ in. four times per revolution of the cam and the number of drops required to lower the ball and stem $7\frac{3}{4}$ in. into the concrete is considered a measure of the consistence of the concrete.

The number of drops required may vary from 20 for very wet concrete (6-in. slump) to 200 for very stiff concrete.

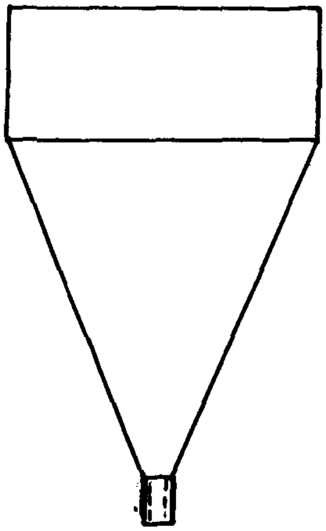

FIG. 9—Flow cone.

It is claimed that the Wigmore consistometer is an improvement over the slump test because work is actually done on the concrete in a way which resembles field conditions. Variations in results may be expected if the ball comes in contact with large aggregate.

Flow Cone:

Contraction joints in dams, cleavage planes in rock foundations, cavities behind tunnel linings, voids in preplaced aggregate, and openings around post-tensioned cables may be filled with grout pumped under pressure. Grouts consist of cement and water or combinations of sand, cement, water, finely divided filler, and admixtures. Grouts must be very fluid to penetrate small cavities. The slump test and other described methods of measuring concrete consistency are unsuitable.

The U.S. Corps of Engineers has prepared a standard test procedure for measuring the flow of grout mixtures by means of the flow cone [22] (see Fig. 9).

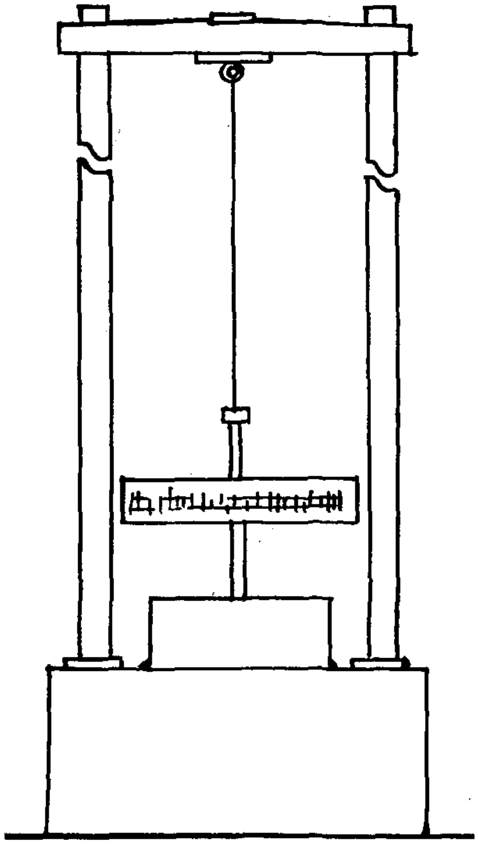

FIG. 10—Grout consistency meter.

This method outlines the procedure to be used in the laboratory and in the field for determining the consistency of grout mixtures by measuring the time of efflux of a specified volume of grout from a standardized flow cone or funnel. The flow cone is mounted firmly with the top surface level, the discharge tube is closed by placing the finger over the end, and 1725 ml of mixed grout poured into the cone. The finger is removed, and the number of seconds until the first break in the continuous flow of grout is the efflux time. When comparing grouts, the speed of mixing and the mixing time have an influence on efflux time and should be maintained constant.

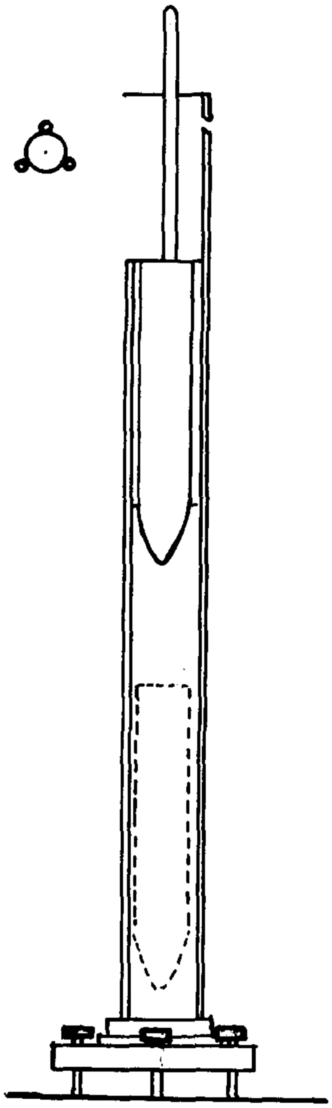

FIG. 11—Otto Graf viscosimeter.

Grout Consistency Meter:

A meter for measuring consistency of grout has been developed at the University of California and is described in the specifications for Restoration of Darker Dam [*23*]. The Grout consistency meter (see Fig. 10) is essentially a torque meter. The sample of grout is placed in a metal pan mounted on a platform that can be rotated at a constant speed of 60 rpm. Suspended from a music wire is a 16-lb paddle assembly to which a torque is applied as the sample of grout is rotated. The angle of twist or consistency factor is read by an index pointer attached to a cross strut.

Otto Graf Viscosimeter:

The Otto Graf viscosimeter (see Fig. 11) has been used in Europe to measure fluidity of grouts. It consists of a brass tube approximately $35\frac{1}{2}$ in. long and $2\frac{7}{16}$-in. inside diameter mounted on a base in an upright position, and an immersion body weighing 5000 g that is $11\frac{3}{32}$ in. long. The immersion body has protruding cams on the sides so that grout can pass between them when the immersion body is placed in the tube containing the grout. In practice the tube is filled to a prescribed level with grout. The immersion body is then placed in the tube and released. The number of seconds required for the body to assume its final position is a measure of the fluidity of the grout.

This equipment was specified as a control measure for grouting operations on a post-tensioned bridge. Consistency was also measured with the flow cone. Tests were made immediately after mixing and again 30 min after mixing. The tests indicate that the flow cone provided the same information as the viscosimeter and was as effective for control purposes. The ratio of viscosimeter reading to flow cone reading was approximately 1:5.

CONCLUSIONS

Concrete knowledge and technology have increased during the years, but additional knowledge will be required if concrete is to maintain the position it has established as the universal building material. New products are being developed in all phases of industry at a rapid rate, and improvements in concrete production, control, delivery, and placing techniques must also be developed. At the same time, more widespread use of available knowledge for controlling uniformity, quality, and workability will improve the competitive position of concrete.

New methods of mixing, placing, consolidating, and finishing concrete may permit the use of much less water and improve concrete quality. Eventually it may be possible to place and consolidate 0 to 2-in. slump concrete in walls, floors, and so on, as readily as 3 to 4-in. slump concrete is placed today. New tests and methods for quality control and measurement of workability must be developed concurrently with methods of mixing and placing concrete. The slump test is an old friend and has served its purpose well. We do not like to see old friends pass away, but change is inevitable. The slump test and other tests used to measure uniformity may be replaced by more efficient test methods in the future.

Meters have been developed to measure moisture content of sand and coarse aggregate in the bins at concrete plants and promote better control of mixing water. Van Alstine [*25*] attributed the uniformity obtained at Denver Reservoir No. 22 Dam, where sand bins were filled 17 to 20 times each day with sand of widely varying moisture content, to the use of an electrical resistance moisture meter. More general use of devices of this type will improve quality control of concrete throughout the industry.

Methods of measuring consistency of concrete while it is being mixed in the mixer drum should be developed so that corrections can be made immediately. The slump test and other tests used to indicate consistency can be made only after the concrete is discharged, and corrections can be applied only to subsequent batches. Polatty [*26*] reported that such a device, called the "Plastograph," gave a better indication of workability than the slump test and indicated a moisture change in time to make a correction in the same batch. The equipment was used at Allatoona Dam with mixes containing 6-in. max aggregate and three bags of cement per cubic yard. When used with 3 or 1½-in. aggregate, the equipment was less efficient.

New methods of controlling uniformity may include determination of water content in freshly mixed concrete by means of neutron moderation and determination of cement content by radio-tracer techniques or electromagnetic radiation methods as suggested by Lorman [*27*]. At the present time these methods are expensive and more suitable for use in the research laboratory than in the field.

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Freshly Mixed Concrete

MAKING AND CURING CONCRETE SPECIMENS

By T. B. Kennedy,[1] Personal Member, ASTM

Synopsis

In the United States the "standard" specimens for strength are the 6 by 12-in. cylinder and the 6 by 6-in. cross-section beams. In many countries cubes are used to measure strength. Both cylinders and cubes have advantages and disadvantages.

The chief purpose of making specimens is to determine the strength of concrete for check of mixture proportions, quality control, indication of time when loads can be applied, and acceptance for payment.

Appropriate ASTM methods cover procedures for making and curing specimens, and these must be followed conscientiously. A major factor among many affecting the proper preparation of specimens is the use of competent personnel. Factors that can have an adverse effect on specimens are the use of nonspecification single-use molds, leaky reusable molds, improper sampling, improper consolidation, inadequate protection of young specimens, improper storage conditions, improper capping, and inadequate curing. Adequate curing becomes more important as the water-cement ratio decreases.

The 1914 Committee Report [*1*][2] formed the basis for the use of the presently accepted cylinders and beams for testing concrete for compressive and flexural strength. S. E. Thompson was chairman of the committee.

ASTM Method of Making Concrete Specimens in the Field (C 31) and Method of Making Concrete Specimens in the Laboratory (C 192) both state: "Compressive test specimens shall be cylindrical, with a length equal to twice the diameter. The standard specimen shall have a diameter of 6 in., if the coarse aggregate does not exceed 2 in. in nominal size." However, provisions are made in both methods for making cylinders of other sizes. Beam size varies widely for specimens cast in the laboratory for experimental testing and can vary in the field, but a beam with cross section of 6 by 6-in. is considered "standard" for the field.

Thaulow [*2*] points out that both cylinders and cubes are used in Europe to measure compressive strength and that neither cylinder nor cube gives a precise picture of the strength of the concrete in the structure, but that the cylinder strength is closer to it than cube strength.

At the RILEM Symposium on Experimental Research of Field Testing of Concrete [*3*] "A RILEM Method of Sampling, Making, Curing, and Strength Testing of Concrete" was proposed for

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

adoption. This method covered fabrication of cubes and cylinders for compression testing and beams for flexure testing; however, a footnote to the method says: "Preference is given to the cube as test specimen for concrete control." The advantage of the cube over the cylinder derives from casting cubes in metal molds with faces machined to required tolerances. This permits them to be turned on their sides as cast and tested without capping. The disadvantages are that cubes are influenced greatly by friction between machine platen and specimen caused by the 1:1 height to base ratio and by the fact that cubes are tested in a direction normal to that of casting. Many investigations, including those on which the correction factors for apparent strength of cores are based, ASTM Method of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42) described by Kesler [*4*], show that end effects are eliminated only when the ratio of height to diameter approaches two. The effect of testing normal to the direction of casting is discussed later.

PURPOSE OF MAKING CYLINDERS OR BEAMS

ASTM Method C 31 speaks of specimens for determining: (*a*) the adequacy of laboratory mixture proportions for strength, (*b*) the basis for acceptance, (*c*) quality control, and (*d*) determining the time when forms may be removed or when a structure may be put in service. Method C 31 deals with ready-mixed or site-mixed concrete in the field. Method C 192 deals with specimens made of laboratory-mixed concrete and obviously is not directly applicable to (*b*) and (*d*).

The report of American Concrete Inst. (ACI) Committee 214 [*5*] states that the primary function of tests of field concrete is to insure production of uniform concrete of desired strength and quality. Concrete, because of its heterogeneity, is subject to the influence of many variables, the effect of which can be measured by the strength of test specimens carefully made under uniform conditions.

The purpose of this chapter is to explain the importance of following the steps in the procedure, to elaborate on the prescribed techniques, and to improve the reader's understanding of the principles involved and the consequences of following or not following prescribed methods. This chapter will be concerned with general conditions and hypothetical considerations that affect good practice and will not repeat clear-cut admonitions stated in the relevant test methods.

FACTORS INFLUENCING THE STRENGTH OF SPECIMENS

Price [*6*], Sparkes [*7*], ACI Manual of Concrete Inspection [*8*], and Walker and Bloem [*9*] are among many who have written about the factors that influence the apparent strength of concrete and, therefore, of strength of concrete test specimens. The following factors all can have a pronounced influence on the apparent strength of concrete specimens: (*a*) compaction, (*b*) specimen molds, (*c*) fabrication techniques, (*d*) temperature of hardening, (*e*) temperature of curing, (*f*) moisture conditions during curing, and (*g*) disturbances during early life. The effect that any of these factors has on the apparent strength of the specimen depends, in turn, on how it contributed toward or prevented the production of the desired test specimen. For example, compaction of specimens of high-slump concrete by vibration might have only slightly adverse effect if the vibration were of very short duration. If prolonged it could cause serious segregation, with consequent weakening of the upper region of the specimen.

Molds that leaked slightly might have no discernible effect on the strength of concrete with less than 1-in. slump molded in them but have a pronounced effect on concrete with 7 or 8-in. slump.

Unless one is deliberately studying the effects of the factors that are involved in making and testing of specimens, everything possible should be done to control these influences so that the variation measured by testing of the specimens will truly reflect variation in the concrete.

SELECTION OF PERSONNEL TO MAKE AND CURE SPECIMENS

Many architects and engineers make the contractor responsible for inspection of his own operations, including the sampling, making, curing, and testing of the concrete specimens to determine compliance with specification requirements. Presumably this practice is believed to save money for the owner. However, the contractor must always pass along to the owner the cost of inspection in the bid price, whether such cost is identified as such or is included in other costs. Naturally, as is true for all aspects of his operations, the contractor attempts to reduce this cost as much as possible, such as by making the minimum number of specimens, frequently by the lowest-paid laborers, in the cheapest molds possible, stored and cured in bare conformance or even nonconformance with applicable test methods, and tested by the laboratory offering to do the job for the least unit cost. The system is wrong. It is like putting the fox in the hen house to guard the chickens.

Wagner [*10*] made a statistical study that indicated lower coefficient of variation and generally high level of strength for cylinders made by qualified laboratory personnel than for cylinders made from concrete from the same source by contractor personnel. Those who sample concrete and make specimens should be carefully chosen, and, insofar as possible, the same persons should make all the specimens for a given project or program. Particular care should be paid to sampling. Above all, the operators should be thoroughly conversant with Methods C 31, C 192, and ASTM Standard Method of Sampling Fresh Concrete (C 172). Their techniques should be observed, and they should be questioned occasionally to determine if they know and observe the provisions in the method.

On request and for a reasonable fee the Cement and Concrete Reference Laboratory of the ASTM and National Bureau of Standards will inspect equipment and techniques of personnel engaged in performing the tests covered in Methods C 31 and C 192.

Suggested minimum reading for personnel making and curing specimens beyond the applicable test methods is the ACI Manual of Concrete Inspection [*8*].

SAMPLING

ASTM Methods C 31 and C 192 both require that samples be obtained in accordance with the provisions of Method C 172. Because the provisions on sampling are in Method C 172 and those concerning the making and curing of specimens are in Methods C 31 and C 192, there is danger that Method C 172 will not be read and observed as carefully as the provisions of Methods C 31 and C 192.

Because sampling has been covered in a separate chapter by Mr. Abdun-Nur, nothing more will be said on this subject than to caution the operator that the batches sampled must be obtained in a completely unbiased manner (do not try to select "typical" batches). The reader is referred to the ACI Manual of Concrete Inspection [*8*], the ACI Committee 214 report [*5*], and the chapter on sampling in this volume for guidance in taking samples.

MOLDING CONCRETE SPECIMENS

The operator should follow the same plan of operation each time he makes specimens. It helps a great deal if he has a helper who prepares the tools, receptacles, molds, and work area ahead of time. This saves the operator's time and helps insure the requirement in Method C 172 that the period between taking and using the sample shall not exceed 15 min is observed.

Tools should be clean and in good condition. Good condition means that the blunted trowel does not have knicked edges, that the blade is straight, that the straight edge is straight to the eye, that the wooden float has a smooth plane surface, and that the pans, buckets, and wheelbarrows are watertight and without large dents that will prevent shoveling or otherwise removing concrete cleanly from them.

Methods C 31 and C 192 speak of blunted trowels. A blunted trowel is made by cutting off its sharp tip on a line normal to the longitudinal centerline of the trowel. A 10-in. brick layer's trowel, from which $\pm 2\frac{1}{2}$ in. have been cut off the tip, is a blunted trowel and a very handy tool for spading along the sides and ends of beams and prisms. Trowels are made of tempered tool steel and thus are difficult, but not impossible, to cut with file or hack saw. Scoring the blade with a sharp file or thin carborundum wheel will permit breaking the point off in a vise. After removal of the tip the edge must be dressed smooth by filing or grinding.

Scoops should have flat bottoms. A D-handle-square-blade shovel is appropriate for remixing the concrete and for handling to beam forms from a flat-bottomed pan or mixing board. Either a square or round pointed shovel can be used for handling concrete into or from a wheelbarrow with a rounded bottom, but a square-end shovel is preferable.

The work area should be adequate in size and, if out of doors, in a place protected from the wind, sun, and weather if at all possible for comfort of the operator and the protection of the fresh concrete.

Beams are sometimes made in wooden molds. Such molds can be made quickly and cheaply, but unless the wood is treated before use by thoroughly impregnating with oil, coating with plastic, or other satisfactory means, they will be absorbent. Wood molds are difficult to make watertight and may warp from absorption of water. Specimens formed in warped molds will undergo eccentric loading during testing with uncertain effects on the results. Occasionally, wood sugars will be leached from boards and react with the cement paste to produce a soft and dusting surface. This has an adverse influence on apparent strength.

Occasionally other materials will be used for molds. The author has seen beams cast in beautifully made, watertight, aluminum molds. The surfaces of the beams were badly etched, and scarred from formation of hydrogen gas resulting from the reaction between the aluminum and the alkalies of the cement. Steel is the best material for beam molds.

Method C 31 permits the use of reusable steel or iron or other metal or plastic molds or single-use molds for cylinders. Single-use molds are the so-called can molds and cardboard molds, and molds of any other material so long as they meet the requirements of ASTM Specification for Single-Use Molds for Forming 6 by 12-in. Concrete Compression Test Cylinders (C 470). Method C 192 does not permit the use of single-use molds. Heavy steel and cast-iron molds are satisfactory for making cylinders of any kind of concrete as long as they meet the requirements for dimensions and are watertight. Plastic molds would appear also to be suitable for general use as long as they meet dimensional tolerances, are watertight, made of a plastic unaffected by the cement chemicals, and maintain dimensional stability while in contact with water, at least within the limits set by Specification C 470.

Molds should be clean inside and out and watertight. If a mold is filled with water and none leaks from its seams and joints it is watertight. There is a feeling that if no visible water runs out of the seams or joints when the mold contains concrete it fulfills the requirement for watertightness. Under the latter criterion a mold could be watertight when it contained a mixture that bled little but not be watertight when used with a bleeding mixture. The best test for watertightness is to fill the mold with water and see if it leaks.

The reason for insisting on watertight molds is that escape of water from the specimen lowers the water-cement ratio of the concrete, thus affecting the strength of the concrete. No two molds would allow the same amount of water to escape, so that the effect of changing water-cement ratio would vary from specimen to specimen. Virtually all unsealed reusable metal molds leak.

Molds can be sealed quickly and easily by use of modeling clay spread with firm finger pressure in a thin ribbon along the outside of seams and joints. The surface to which the clay is applied must be clean and free from oil. Thin ribbons of clay extruded from a slit or small hole in the cylinder of a homemade pipe and plunger apparatus are satisfactory.

Molds should be lightly oiled before use to keep the concrete from bonding to the mold. A thin but continuous film of oil does just as good a job as a thick coat. Neither Method C 31 nor C 192 specifies the oil to use beyond saying that it shall be mineral oil. SAE 20 or 30 automobile motor oil is satisfactory. The U.S. Bureau of Reclamation (USBR) [*11*] recommends soft (No. 2) graphite grease.

The sheet metal can molds are suitable if they do not leak and if they meet the dimensional requirements of Specification C 470. A split steel sleeve that can be slipped over the outside of the can, plastic, or cardboard mold, then tightened up snugly against the mold, will provide support and dimensional stability while the cylinder is being made.

The USBR [*11*] recommends that the can be placed on a smooth wooden or metal disk of the correct size to fill the recessed space in the bottom of the can during the filling and consolidation process to protect the bottom and prevent the formation of convex ends on the cylinders.

Removal of the can should, of course, be done carefully so as not to damage the specimen. Cans usually may be peeled away by cutting a nick at the seam with metal snips, then opening the can along the seam by pulling apart with two pairs of pliers. The author has seen cans removed by grasping the can with a pair of pliers at the seam, then beating can, specimen, and all on the ground until something gave way. This is bad practice and should be avoided.

If cardboard molds are used, they should be purchased under the requirements of Specification C 470 and tested for compliance. Many cardboard molds do not comply. Cardboard molds that do not meet the absorption and volume stability requirements of Specification C 470 may swell slowly on absorption of water from the concrete and put the weak, partially hardened concrete under sufficient tensile stress to cause micro or visible cracks and appreciably weaken the specimens. The author has seen specimens cracked so badly by expansion of cardboard molds that they came in two when the molds were removed. Some agencies, for example USBR [*11*], do not permit the use of cardboard molds. Burmeister [*12*] compared the strength of cylinders cast in steel molds with those cast in paper molds from two sources. He found the loss of strength averaged 5.6 per cent with molds from one source and 12.3 per cent with molds from the other. Molds from neither source would have met Specification C 470.

If cardboard molds are used, it is abvisable to keep water away from their outside surfaces. Wet burlap or other wet materials should not touch the outside of the molds. The molds should not be buried in wet sand until the concrete is thoroughly hardened. These admonitions are given because the outside surfaces of the mold may not be waterproofed as thoroughly as the inside so that contact with water will cause the mold to swell, with undesirable consequences.

Tools, before use, should be dampened and covered with wet burlap or other wet but not dripping cloth, but the surfaces of the tools and receptacles should not have free water on them.

The sample should be inspected carefully for visible evidence of segregation. This, if present, should be corrected in the remixing.

The sample of concrete (note that Method C 172 says the sample shall be not less than 1 ft^3) should be covered with a wet but not dripping cloth, and uncovered only during the remixing before the specimens are molded. During the molding process, only a sufficient area of the sample should be uncovered to enable the operator to remove the necessary portion for the specimens.

In handling concrete the shovel or scoop should be turned 180 deg just as it enters the mold so that the back of the shovel or scoop is up or toward the mold as the concrete leaves it. This keeps coarse aggregate from segregating and presents a mortared surface to the form wall as the concrete slides from the shovel or scoop.

If the concrete is of soft enough consistency to require rodding, the strokes should be counted carefully and the rod should not strike the bottom of the mold. Impact of the rod can damage the mold.

The objective of rodding or vibrating concrete after placement in the molds is to produce a specimen that is properly compacted and homogeneous throughout. That is why cylinders that are to be rodded are made in three layers and those to be vibrated in two. That is also the reason both Methods C 31 and C 192 contain rather explicit instructions on how to rod and how to vibrate. Both Methods C 31 and C 192 in specifying how to rod state that the rodding shall be throughout the bottom layer, and for each upper layer the rod shall penetrate about 1 in. into the underlying layer. Some operators, especially when rodding soft concrete, force the rod clear through all three layers. This causes segregation. It tends to push the coarse aggregate to the bottom and soupy mortar to the top. It defeats the purpose for which the detailed instructions were put in the methods, and should not be done.

All holes left in the concrete when the rod is withdrawn should be closed by tapping the sides of the mold. A 1-lb rubber hammer or wood mallet of about equal weight is suitable for tapping heavy-steel and cast-iron molds. The flat side of the blunted trowel can be used for tapping can, plastic, or cardboard molds. Any voids left from rodding will weaken the specimen. Molds should be tapped only sufficiently to close the voids. It is not advisable to tap single-use molds with the tamping rod, because this may dent the mold.

Concrete of slump between 1 and 3 in. may be rodded or vibrated. If the slump is less than 1 in. the concrete must be vibrated, and it will usually be advantageous to vibrate normal weight concrete with slump less than 3 in. Lightweight aggregate concrete of quite low slump is usually readily consolidated by rodding. Either external or internal vibration judiciously used will give good results. There will be no difference in strength between specimens consolidated to the same density by rodding or vibration providing the method of consolidation does not produce areas of segregation.

Data obtained in a cooperative study involving six laboratories before provisions for vibration were put in Methods C 31 and C 192 generally indicated higher strength for specimens of stiff consistency when vibrated than when rodded. This effect seems to be more pronounced with lean mixtures than rich [*13,14*]. The author's preference is for internal vibrators. They can be carried to the work, they usually operate at higher frequency than platform vibrators, and the molds do not have to be fastened to the platform. When using an internal vibrator one should be sure to observe the precautions about vibrator diameter. A vibrator with diameter of 0.75 in. is permitted, but one with head of 1 in. or slightly larger will usually do a better, quicker job. If a platform vibrator is used, the device should be inspected to see that the driving belt is tight and that the eccentric weights cannot slip, or, if the vibrator is powered electromagnetically, that the same rheostat setting is used each time so as to induce the same amplitude of vibration.

The important considerations in using vibration are knowing how and how much vibration to apply. Both Methods C 31 and C 192 give detailed instructions as to how to apply vibration to cylinders and beams, but the duration of vibration is sometimes difficult to judge. A good operator will quickly develop judgment based on experience. Excessive vibration can cause segregation, especially if the concrete is oversanded. Excessive vibration can substantially reduce air content, both entrapped and entrained [*15*]. The objective is to apply enough vibratory energy to eliminate all internal voids not contemplated in the proportioning computations, but not so much that the entrained-air bubbles are driven out. The latter is indicated by accumulation of a frothy, watery layer of mortar at the surface. The optimum duration of vibration depends on the effectiveness of the vibrator and the slump of the concrete [*11*]. In vibrating cylinders, three insertions of 3 or 4 sec each per layer are usually enough. Vibration is considered adequate when the top surface exhibits a shiny wet appearance. The Corps of Engineers [*16*] procedure requires that the concrete be consolidated sufficiently to remove entrapped air and prevent honeycomb, and states that usually sufficient vibration has been applied when the surface of the concrete becomes relatively smooth and has a glazed appearance.

If one visualizes vibration as a process that converts the plastic concrete into a heavy fluid, then the end point for application of vibration will be reached just as soon as the liquid completely occupies the mold, which is usually a matter of a few seconds.

If a series of specimens is being prepared by vibration they should be located on a solid base, or immediately after consolidation they should be removed to a vibration free location so that as the series is made those that were consolidated first do not pick up stray vibrations as subsequent specimens are consolidated. Stray vibration can accentuate bleeding with possibly poorer bond between underneath surface of aggregate particles and mortar and noticeably poorer strength. This is not to be confused with revibration which is a vigorous process that rearranges the aggregate particles and further consolidates the concrete, thereby generally increasing the strength.

After consolidation, cylinders should be struck off with a trowel or straightedge. Beams are best struck off and finished with a float. The operator should not overfinish, but obtain a true level finish then quit. Although both Methods C 31 and C 192 permit the specimens to be covered after finishing with glass or metal plates, damp burlap, or other coverings to prevent evaporation from the concrete, in practice it is difficult, especially with beams, to cover them with wet burlap without doing a little damage to the newly finished surface. Also, wet burlap becomes dry in an hour or less under conditions of low relative humidity. Experience has shown that covering the specimens with metal or glass plates, covered by wet burlap, which is in turn covered by a sheet of polyethylene weighted to the ground all around the edges, is an extremely effective means to prevent escape of moisture.

Methods C 31 and C 192 require that cylinder molds sit upright on their bases with the top open to receive the concrete. This provision makes it necessary to mold cylinders in an upright position. Methods C 31 and C 192 do not forbid, but certainly do not contemplate, that cylinders may be turned on their sides before they have hardened. Turning cylinders on their sides causes the bleeding to occur in a direction perpendicular to the axis of the cylinder. The turned-up side will be less dense and strong than the bottom side, and specimens so made can be considerably weaker than specimens allowed to harden upright.

Wright [*17*] in discussing discrepancies in strength results on similar specimens obtained with different testing machines described tests made on concrete cubes in Great Britain. The cubes were made in machined molds in upright position and tested on their sides. The cubes were tested in machines with spherically seated platens and hydraulically self-aligning platens. Those tested under hydraulically aligned platens were significantly weaker. This is attributed to unavoidable characteristics of the method of making the cubes (top as cast, is weaker), and the fact that the hydraulically aligned platen permitted the specimen to strain unequally, thus permitting the weaker edge to reach its ultimate strain capacity and failure load faster than when tested under an unyielding platen that tended to cause the cube to strain more evenly.

Cylinders turned on their sides before hardened, or cast in molds with ends closed by steel plates to eliminate need for capping, would be expected to be weaker along the upper surface as cast, to a variable degree depending largely upon the slump and bleeding characteristics of the concrete. Such specimens could test weaker than those cast and hardened in an upright position, especially when tested in machines with hydraulic or extremely well lubricated platens that freely followed the tendency of the specimen to strain in accordance with its varying modulus of elasticity.

CAPPING

Capping should be considered an operation in the making of a concrete specimen. Capping provides a plane true surface for the application of the load from the testing machine to the test specimen. Capping is accomplished by the application of a suitable material to the ends of cylinders, under the bearing points of beams, if needed, to the faces of "modified cubes," and so on. Much has been written about capping, its advantages and disadvantages, materials that are appropriate, and so on.

If test cylinders are made in rigid molds with an appropriately machined bottom and if they are properly capped with neat cement paste on the top end before they have hardened, they will not require further capping before testing. Johansen [*18*] found the strength of such cylinders when capped 2 to 4 hr after molding equal to the strength of cylinders with the ends ground plane before testing. But he found that caps applied to low slump and to rich concrete, if too thin, cracked and frequently warped. He found that cap thickness should be about $\frac{1}{8}$ to $\frac{1}{4}$ in. Strength of concrete he tested ranged from approximately 1500 to 6300 psi.

Some laboratories possess equipment for grinding and lapping the ends of specimens to the required planeness. This is an excellent, but usually expensive procedure. Attempts to grind the ends of specimens by hand are usually unsuccessful and frustrating. Even though somewhat expensive, grinding insures that weak unsuitable materials are not applied to the ends of cylinders before testing. This becomes increasingly important as the strength of the concrete increases.

The ideal capping material is one that matches the strength, Poisson's ratio, coefficient of friction, and Young's modulus of the specimen on which used.

Plaster of paris alone or mixtures of plaster of paris and neat cement are frequently used. They are cheap, but weaker than practically all concrete made today and should not be used.

High-strength gypsum plasters are available and are satisfactory for most concrete when properly prepared with the water content recommended by the manufacturer to produce the maximum strength.

Sulfur-silica mixtures are popular for capping, but they can vary in properties [*19,20,21*], and should be checked from time to time. Troxel [*22*] showed that end condition of the cylinder under the cap influenced apparent strength. Ends not normal to the axis of the cylinder, or ends that were concave or convex under the cap, caused drastic reductions in strength of cylinders capped with plaster of paris. The adverse effect was much less with cylinders capped with high-strength gypsum plasters or with a sulfur-silica compound.

Gaynor and Wedding [*21*] found little difference in the apparent strength of nominal 5000 psi (strength at 28 days) cylinders when capped with neat cement (applied 3, 7, or 21 days before testing), or sulfur-silica $\frac{1}{8}$ or $\frac{3}{8}$ in. thick. Caps of plaster of paris $\frac{1}{8}$ and $\frac{3}{8}$ in. thick reduced the strength about 4 and 14 per cent, respectively.

Their cylinders were all made in steel molds with machined bottom base plates; therefore, only the top end of each cylinder required capping. They found that increasing the temperature at which the sulfur-silica mixture was used from 245 to 335 F decreased the strength of 2 by 4-in. cylinders made of it from about 8630 to 6645 psi.

The increasing use of high-strength prestressed and precast building elements makes it more important for the laboratory to properly prepare the ends of specimens for testing. Unpublished data in the author's laboratory indicate that confining high-strength gypsum plaster or sulfur-silica caps in heavy steel rings may afford adequate end preparation for concrete with strength exceeding 10,000 psi.

CURING SPECIMENS

The ACI Manual of Concrete Inspection [*8*] says, "By 'curing' of concrete is meant keeping it moist and, if necessary, warm so that hydration of the cement can continue."

Deliberate curing, as distinguished from incidental curing brought about by rain, moisture in the subgrade, periods of extremely high relative humidity, and so on, is effected by providing a favorable moisture and temperature environment, within the limitations of the test methods in which the hydration reaction can proceed. Hydration is a chemical reaction between the cement and water and, like other chemical reactions, proceeds more rapidly at higher temperatures and virtually stops if a supply of water is not maintained. Powers [*23*] says that the amount of water required to hydrate cement is somewhat irrelevant to the question of proper curing. Curing procedures should be such as to keep the concrete as nearly saturated as possible until the originally water-filled capillary space has become filled with hydration products to the desired extent. For maximum hydration the concrete must be kept saturated because hydration products form only in the water-filled capillaries in the cement paste. Loss of water from the paste for any cause reduces the water-filled space available to accommodate hydration products. As concrete hydrates water is removed from the capillaries by the hydration reaction (the cement and water combine chemically) and self-desiccation occurs. Its effect is to slow down and eventually stop the reaction. Hence, portland cement can undergo optimum hydration only if a sufficiently high degree of saturation of the paste is maintained to keep the capillaries full or nearly full of water. The effect of self-desiccation is felt more severely with richer than with leaner concretes. The reader is urged to study the article by Powers [*23*] and an article by Powers, Copeland, and Mann [*24*] for an interesting and instructive treatment of the mechanism and effects of cement hydration.

The cement-water relationships that influence hydration dictate conditions of humidity that should be maintained during the curing of concrete test specimens, but temperature also influences apparent strength. Concrete permitted to harden at 50 F, then cured at 70 F till time of test, will be stronger than similar concrete that hardened and cured at 70 F, which in turn will be stronger than concrete hardened at 90 F and cured at 70 F. Concrete hardened at 70 F and cured at 90 F will be stronger at 28 days than concrete hardened at 70 F and cured at 70 F, and this concrete will be stronger than concrete hardened at 70 F and cured at 50 F. Price [*6*] shows strength of 28-day cylinders cast at 40 F and cured at 70 F was about 700 psi higher than similar concrete cast at 115 F and cured at 70 F, and specimens cast at 40 F and cured at 40 F were only about 78 per cent as strong at 28 days as those cast and cured at 70 F. Probably more damage is done to strength by excessively high temperature during the early stages of curing than by low temperature.

Curing begins as soon as a specimen is struck off and continues until it is broken in the testing machine. During this period everything possible should be done to prevent its drying even to the slightest extent, and everything possible should be done to maintain the curing temperatures within the range set forth in the test method or specification. This admonition also applies while cylinders or beams are being transported from the job site to the laboratory. During this period they should be carefully packed in damp sawdust or sand and preferably put into a thermally insulated box. They should never be allowed to bounce around in the back of a pickup truck or be exposed to the elements.

Although both Methods C 31 and C 192 make provision against it, some laboratories allow specimens cast in cardboard molds or in sheet-metal cans to stay in their containers on the fog-room shelf until time of test. This procedure is not satisfactory because the specimens are sealed against the entrance of curing water, and it is particularly undesirable for concretes with water-cement ratios less than 0.5 by weight. Price [6] stated that water-cured specimens with water-cement ratios of 0.55 were about 10 per cent stronger at 28 days than those cured in a fog room at 100 per cent relative humidity. Walker and Bloem [9] indicate that even slight drying lowers flexural strength. When beams are permitted to dry partially, the effects of self-desiccation and tensile stress due to localized drying combine to lower the apparent strength.

Sometimes it is assumed that if the relative humidity in a fog room is near 100 per cent the environment is necessarily suitable for all curing purposes. This is not true because specimens may dry to some extent unless free water is maintained on all surfaces of the specimens at all times as required by Methods C 31 and C 192. Because of the effect even of slight drying, especially on the flexural strength, both Methods C 31 and C 192 require beams to be cured for the last 20 hr before testing immersed in saturated limewater. The author feels that all low water-cement ratio concrete should be cured by storage in saturated limewater. Then one is sure that "free water is maintained on all surfaces of the specimens at all times" and that self-desiccation will not slow down the hydration process.

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STP169A-EB/Apr. 1966

Freshly Mixed Concrete

SETTING TIME

By T. M. Kelly,[1] Personal Member, ASTM

Scripture's paper on Setting Time [*1*],[2] published in 1955, summarized the problems involved and the progress made, as of that time, in developing methods for measuring the setting time of concrete. He pointed out that the setting time of cement does not adequately define the setting time of concrete and warned that very misleading conclusions may result from assuming such to be the case. Many criteria for defining setting time of concrete had been suggested, including loss of workability before placing, impairment of bond of freshly placed concrete to concrete in place, time of finishing for floors, ability to strip forms, and early strength gain. Scripture was of the opinion that none of these criteria represents the setting time of concrete, stating that as soon as concrete has any appreciable strength it has certainly set, and conversely, as long as concrete maintains any significant degree of workability, it is not set. He concluded that one or more points between these two extremes represent the setting time of concrete and, like the setting time of cement, will necessarily have to be defined by a particular test method and apparatus. Subsequent investigations of the rate of hardening of concrete have tended to substantiate these conclusions.

Methods of Test

The various methods of test for determining the setting time of concrete which had been proposed or investigated prior to 1955 were described by Scripture, and their merits were discussed as summarized below:

Electrical Measurements:

The electrical measurement method [*2, 3*] consisted of measuring the change in electrical resistance of concrete mixtures with time. For a concrete mixture without any additive, the electrical resistance was found to increase fairly steadily until it reaches a point at which the curve leveled off. A similar concrete mixture containing calcium chloride, however, had an almost constant and very low electrical resistance throughout the period of test. It appeared, therefore, that such a test method would be very limited in its application and would be of little use where it is desired to show the effects of chemical admixtures on the setting time of concrete.

Consistency Measurements:

Apparatus suggested for use in relating change in consistency with setting time were the Vebe apparatus, modified Gillmore and Vicat needles, penetration rods, and the Kelly ball. The penetration methods, in general, were considered to be unsatisfactory because of interference of large pieces of aggregate. Penetration tests, if applied to mortar sieved through

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

a No. 8 sieve, gave more reproducible results than those obtained on neat cement paste, but doubt was expressed that the results really represented the time of setting of concrete as a whole in view of the discrepancy between the setting times of neat cement and mortar pats.

Troweling Method:

A method based on determining the point at which it is just possible to finish the surface of the concrete by steel troweling was described as giving reasonably reproducible results, though somewhat subject to personal factors. It was considered questionable whether this method measures the setting time of concrete for those interested in anything except floor finishing.

Velocity and Frequency Measurements:

The soniscope method [*4*], based on measurement of wave velocity, was considered to have application as a research tool but was cited as being too high in cost and requiring too delicate a technique for consideration as a standard test method. Results obtained using conventional sonic apparatus to measure fundamental frequencies and employing a mix confined in a metal tube were considered sufficiently promising to merit further investigation.

Bleeding Characteristics:

This method had been suggested but no data were available on its application to the determination of setting times. Because of the wide variation in bleeding characteristics of different concrete mixtures, the proposed method was not considered promising.

Heat of Hydration:

Though curves obtained by this method showed sharp changes where the setting time of cement would be expected, its applicability in determining the setting time of concrete was doubted on the basis that the method measures a chemical property of cement and that the shape of the curve could be completely changed by modifying the gypsum content of the cement without actually changing the setting time of the cement or concrete.

Changes in Volume:

Determination of the setting time of concrete from measurement of changes in volume of the setting concrete in a dilatometer was considered to be open to the same objections as those stated in connection with heat of hydration determinations.

Strength Determinations:

One method based on strength determinations consisted of casting specimens in flexible containers and determining deflection. Some work had also been done on both compressive and flexural strength determinations, but sufficient reliable data were not available to demonstrate the suitability of any of these methods for measuring setting time. From limited results obtained by a tension test method it appeared that this method showed some promise, since a fairly sharp break in the curve appeared to occur at a definite time.

Deformability Changes:

The pressure-type air meter had been employed to determine changes in deformability of air-entrained concrete. Curves in which percentage of air was plotted against time after mixing showed very definite points of inflection when the concrete became sufficiently rigid to resist deformation by the air pressure commonly used in this type of meter. Such a method would, of course, be applicable only to air-entrained concrete.

Scripture tentatively concluded that probably more than one definition of setting time would be needed, since setting time of concrete means something different to each individual, depending on the specific application and that setting time would probably be defined in terms of a maximum change in some physical rather than chemical property of the concrete, determined by a particular test method. Any definition, therefore, would necessarily await the establishment of a suitable test method.

Penetration Resistance Method:

Substantial progress has been made in measuring the rate of hardening of concrete subsequent to publication of the 1955 paper on Setting Time. Tuthill and Cordon [*5*] developed a method based on the use of Proctor penetration needles to determine the hardening characteristics of concrete mortar sieved from concrete. Their work and application of their method by others indicates that correlation can be established between the hardening of concrete and of the mortar sieved from it. The authors state, "Results of tests indicate that this equipment provides an accurate, rapid, and economical method of determining hardening characteristics of concrete mortar which, though not equal to those of corresponding concrete, are of similar character and reliably indicative of what may be expected of the concrete." In their test procedure the mortar was sieved from the concrete and placed in a container at least 6 in. deep, the container being large enough to permit a minimum of ten undisturbed readings of penetration resistance. After the mortar had been vibrated in the container, it was covered and placed in a room maintained at the desired temperature of test. Bleeding water was poured off before making a penetration test. Penetration resistance readings were taken at such intervals as necessary to define the hardening characteristics by means of penetration needles of appropriate end areas. The first reading was taken with a needle having a 1-in.^{2} bearing area, and subsequent readings were taken with this needle until penetration resistance reached or approached the maximum capacity of the spring. Smaller needles were used in subsequent readings, the size being dictated by the penetration resistance of the mortar and the maximum capacity of the spring. In making penetration resistance readings the pressure on the needle was applied steadily and gradually until the needle had penetrated the mortar to a depth of one inch. In conjunction with penetration resistance tests, a $\frac{1}{2}$-ft^3 container was filled with concrete for observation of its hardening progress and its response to vibration during the period of penetration tests of its mortar, and a concrete test cylinder was made from the same concrete for determination of compressive strength of the concrete when the penetration resistance of its mortar reached 4000 psi. A second concrete cylinder was made for test at 48 hr. The authors stated that these tests were made to provide further information on the early hardening characteristics and to give confidence that the retarded concrete would proceed to develop proper strength.

Tuthill and Cordon defined the vibration limit of concrete as that point during hardening of concrete when it no longer can again be made plastic by revibration. That is, it is a point beyond which a delayed second layer of concrete will not become monolithic with its preceding layer. They stated that a running vibrator will not sink of its own weight into concrete that has passed the vibration limit and that, as measured by resistance of the concrete mortar to the penetration test, it has been demonstrated that the vibration limit will be reached at a resistance of approximately 500 psi. They indicated that a mortar was considered completely hardened when its penetration resistance reached 4000 psi, at which time the same concrete from which the penetration resistance mortar test specimen had been made had developed a compressive strength, determined on a 6 by 12-in. cylinder of approximately 100 psi.

Subcommittee III-n on Setting Time of Concrete, ASTM Committee C-9, subsequently proposed a test method based on the procedure developed by Tuthill and Cordon. This method has undergone many revisions since it was first adopted and is presently entitled, ASTM Test for Time of Setting of Concrete Mixtures by Penetration Resistance (C 403 – 65 T). In scope this specification provides a procedure for determining the time of setting of concrete with a slump greater than zero by testing mortar sieved from the concrete mixture. The definitions of times of setting are admittedly arbitrary, since the hardening of concrete is a gradual process. Under this method times of initial and final setting of concrete are determined on the basis of a rate of hardening test made by means of penetration resistance needles on mortars sieved from the concrete mixture. The times of setting are determined from rate of hardening curves obtained from a linear plot of rate of hardening data, with elapsed time as the abscissa and penetration resistance as the ordinate. This specification defines setting times as follows: (*a*) time of initial setting—the elapsed time, after initial contact of cement and water, required for the mortar sieved from the concrete to reach a penetration resistance of 500 psi and (*b*) time of final setting—the elapsed time, after initial contact

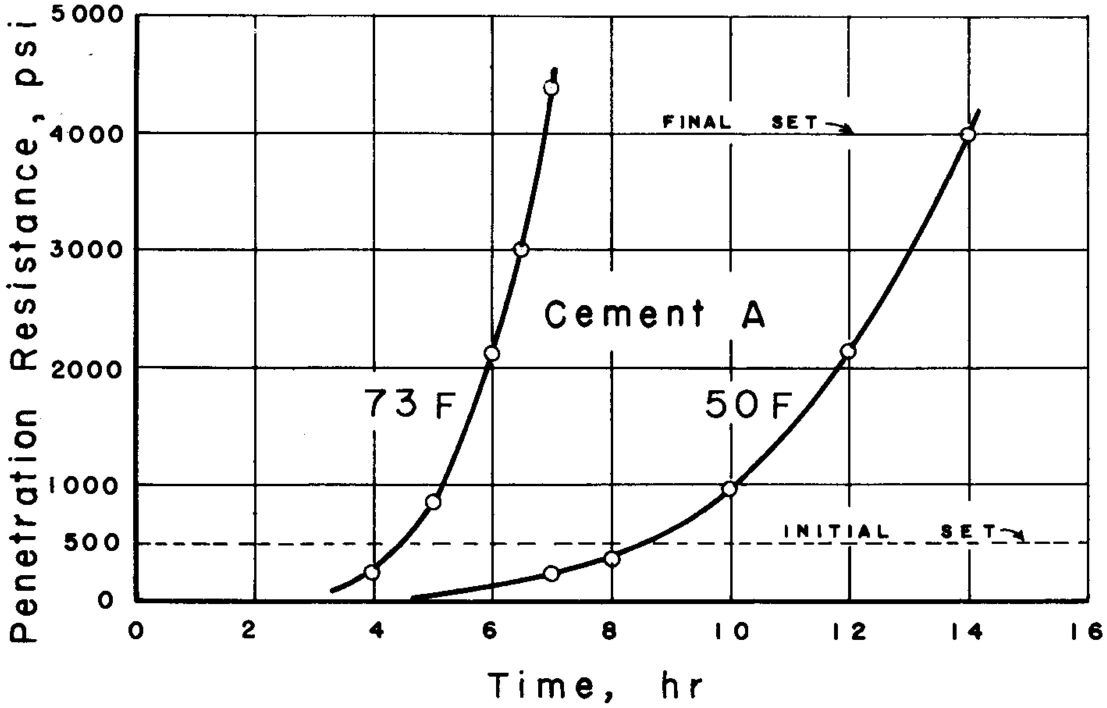

FIG. 1—Effect of low temperature on setting time.

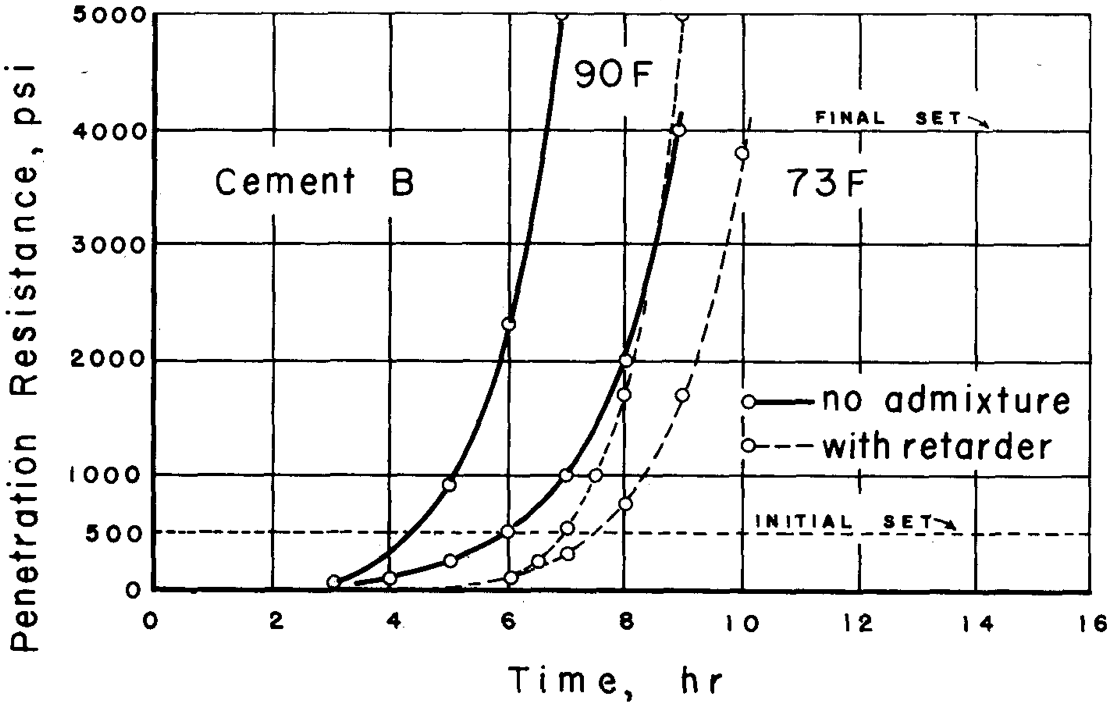

FIG. 2—Effect of temperature and a retarder on rate of hardening.

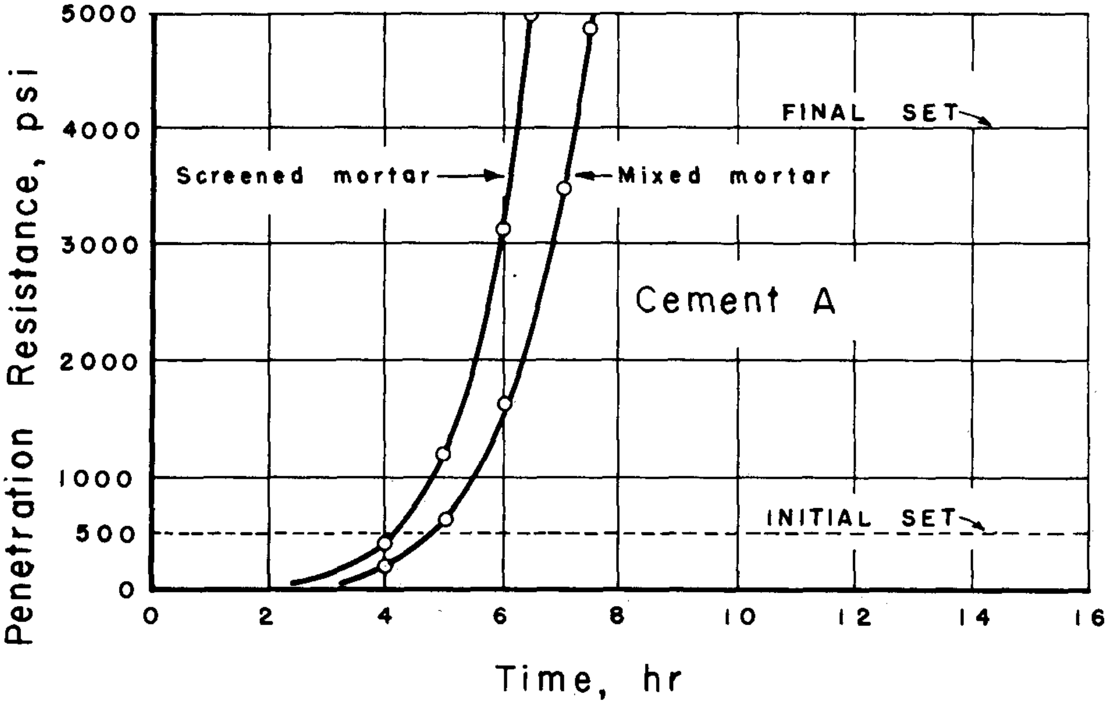

FIG. 3—Rate of hardening of screened versus mixed mortar.

of cement and water, required for a mortar sieved from the concrete to reach a penetration resistance of 4000 psi.

It is apparent that the above definitions of times of setting are based on Tuthill's and Cordon's vibration limit and the penetration resistance at which they considered the mortar to be hardened. Users of the test method are not in complete agreement that 500 psi is, in fact, the vibration limit or that mortar should be considered to be completely hardened at 4000 psi penetration resistance. Such agreement, however, is not essential. These arbitrarily defined points serve as convenient reference points for determining the relative rates of hardening of mortars from different concretes during both the early and later stages of hardening and for determining the effects of variables such as temperature, type and brand of cement, cement factor, mixture proportions, slump, additions, and admixtures upon the time of setting and hardening characteristics of the mortar. Where the test method is being used as a part of performance specifications, these arbitrarily defined points also make it simpler to specify time of setting requirements. It should be kept in mind, however, that the entire body of data from a rate of hardening test provide much information of value which may not be apparent if only the initial and final setting times are reported. Changes in the rate of hardening during the hardening period can be of considerable interest and value in scheduling concrete placing and finishing operations.

Typical rate of hardening curves obtained by the penetration method are shown in Figs. 1–4. The rate of hardening tests were made in accordance with ASTM Method C 403 – 65 T, except that each curve represents a single rate of hardening test, rather than an average of three or more. Figure 1 demonstrates the effect of a low-storage temperature on rate of hardening of mortars sieved from two concrete mixtures. The concrete mixtures were both batched and mixed at approximately 73 F and were similar in all respects, except that after mixing and sieving to provide mortar for penetration tests, mortar specimens from the two mixtures were stored at 73 and 55 F, respectively. Initial and final setting times of the mortar were

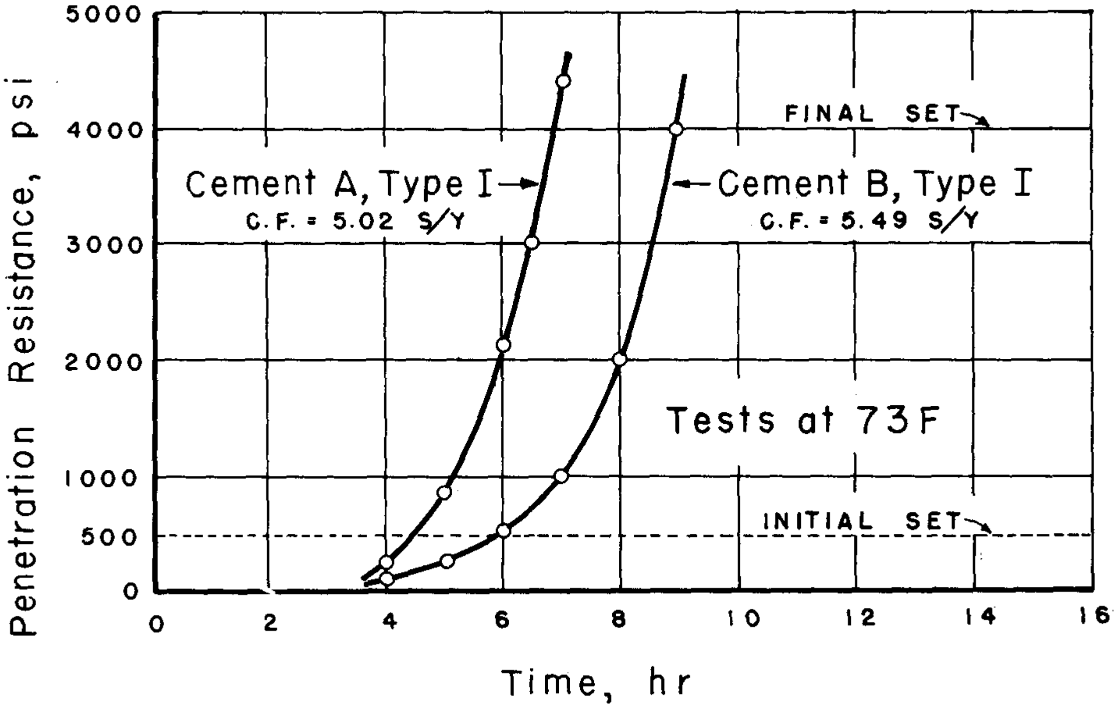

FIG. 4—Effect of brand of cement on rate of hardening.

retarded approximately 4 and 7 hr, respectively, by the lower storage temperature. Retardation of initial set probably would have been greater if the mortar specimens for low-temperature storage had been at 50 F at the start of the test. Figure 2 demonstrates the effects of a retarding admixture at two storage temperatures, namely, 73 and 90 F. Temperature of all of the mixtures after mixing was approximately 75 F. The concrete mixtures were similar in all respects except that concretes containing the admixture, which was added at the rate of 0.266 per cent by weight of cement, were reproportioned to maintain a constant cement factor. After sieving mortar from the concrete mixtures, companion mortar specimens from the two types of mixtures were stored at 73 and 90 F, respectively. With this particular cement and admixture, and mortar temperatures at 75 F at the start of test, the retarding effect of the admixture was greater at the higher storage temperature. In Fig. 3 the rate of hardening of a mortar sieved from a concrete mixture is compared with the rate of hardening of a separately mixed cement:sand mortar. Cement of the same brand and type, and sand from the same lot were used in both the concrete and the mortar mixtures, and the water-cement ratio and cement:sand ratio were the same for both mixtures. Tests on the two mortars were conducted simultaneously. Initial and final setting times were approximately $\frac{1}{2}$ and 1 hr greater, respectively, for the separately mixed mortar. Figure 4 illustrates differences in the rate of hardening of concretes made with two different brands of Type I portland cement. The concretes contained sand and coarse aggregates from the same sources, and ambient temperatures were the same during the mixing and testing period. Though the concrete made with Cement B had a higher cement factor than that made with Cement A, it reached final set approximately 1 hr later.

Bond Pullout Pin Method:

In 1957 a paper by Kelly and Bryant [*6*] was published describing a bond pullout pin method for measuring the rate of hardening of concrete. In this test procedure the whole concrete was utilized, rather than the mortar portion, and the method was based on measurement of the rate of development of concrete bond strength. Stainless steel pins, $\frac{3}{8}$-in. in diameter, were embedded vertically in concrete to a depth of 5 in. by vibrating the concrete around them in a beam mold immediately after the concrete was mixed. The pins were held firmly in a vertical position by a special jig during specimen fabrication. Individual pins were subsequently pulled out of the concrete at increasing time intervals by means of a spring scale and the load at bond failure measured. Rate of hardening curves were obtained by plotting bond stress against time on a linear scale.

The authors stated that in the hardening process no sharp breaking point occurs which can be interpreted as time of setting. Instead, concrete hardens at a gradually increasing rate until it reaches a definitely hard or set condition. On the basis of the observed relationship between time and the degree of hardening of concrete, they were of the opinion that a test method to measure setting time *per se* is not feasible. They believed, however, that the rate of hardening of concrete under any given set of conditions can be determined and that setting times can be arbitrarily defined on the basis of such rate of hardening determinations. Subsequently, a proposed tentative test method was drafted for consideration by Subcommittee III-n, ASTM Committee C-9, in which the test procedure was essentially the same as that de-

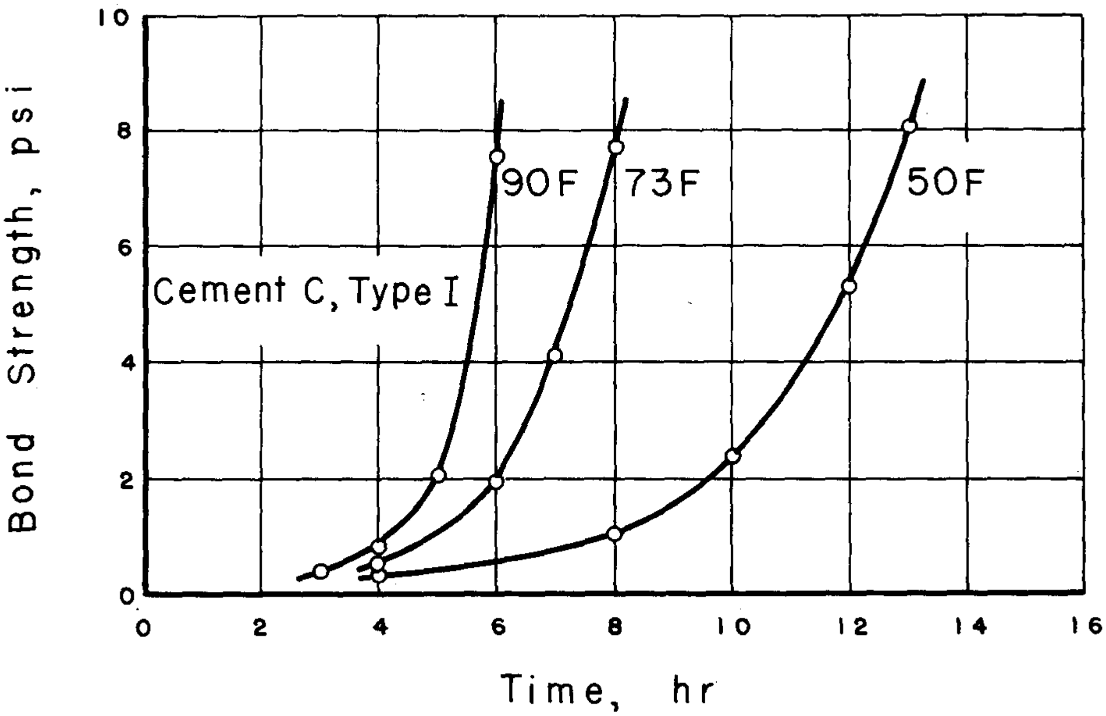

FIG. 5—Effect of temperature on rate of hardening.

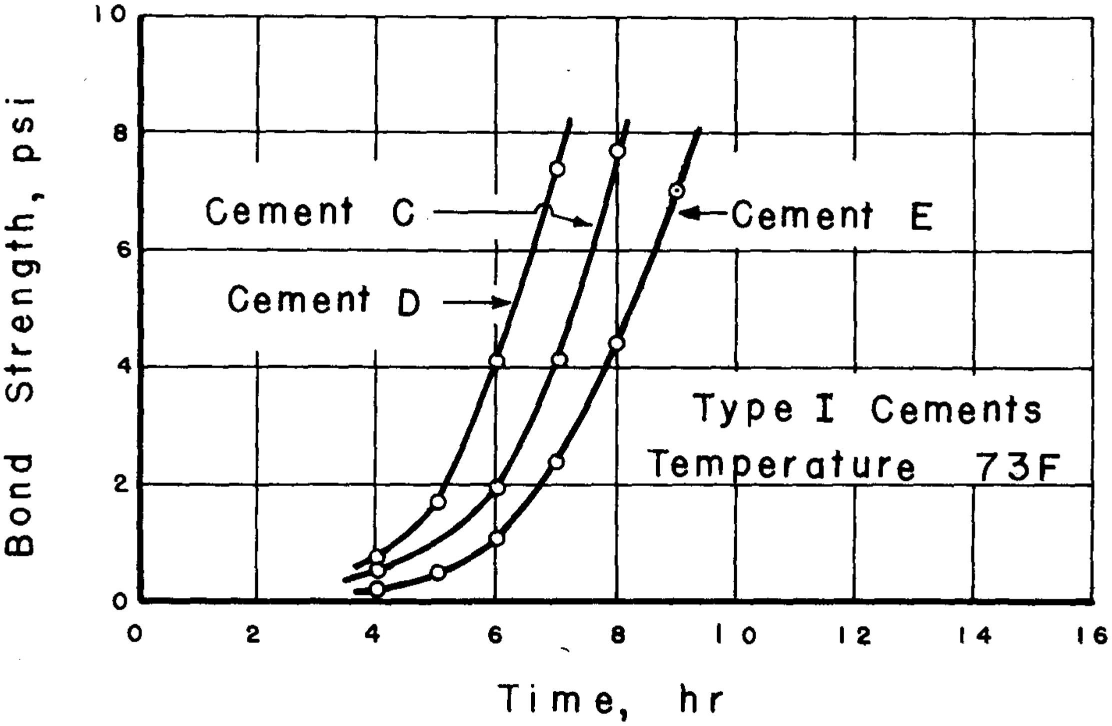

FIG. 6—Effect of brand of cement on rate of hardening.

scribed in the above paper, except that the depth of pin embedment was increased to 6 in. to permit the use of a conventional 6-in. beam mold. This method is under consideration by Subcommittee III-n as an alternate method for determining time of setting of concrete.

A favorable feature of the bond pull-out pin test method is the fact that the rate of hardening determinations are made on the whole concrete, rather than on the mortar sieved therefrom. The results, therefore, can be interpreted without concern as to the possible effects of removal of coarse aggregate or of manipulation and exposure of the sample during the process of sieving the concrete to obtain mortar for the penetration tests.

On the other hand, modification and refinement of the method are desirable. For example, the relation between the embedded surface area of a pin and combined weight of beam and specimen are such that the maximum load is limited, unless the specimen is physically restrained from being lifted during the later stages of hardening. Preliminary studies indicate that higher bond stresses may be reached by reducing pin diameter or reducing depth of embedment or both.

Typical rate of hardening curves obtained by the bond pullout pin method are shown in Figs. 5–7. All of the concrete mixtures for which rate of hardening curves are shown were similar in all respects, except for differences in brand of cement used or the addition of a chemical admixture. The temperature of the mixing room was maintained at approximately 73 F, and all concrete mixtures were at approximately the same temperature upon completion of mixing. Immediately after their fabrication, the rate of hardening specimens were stored for the duration of the tests at the temperatures indicated on the charts. Figure 5 illustrates the effect of different storage temperatures on rate of hardening. Figure 6 illustrates the differences in rate of hardening that can result from a change in the brand of Type I cement being used in a given concrete mixture. Figure 7 shows the rate of hardening at various storage temperatures of a reference mixture

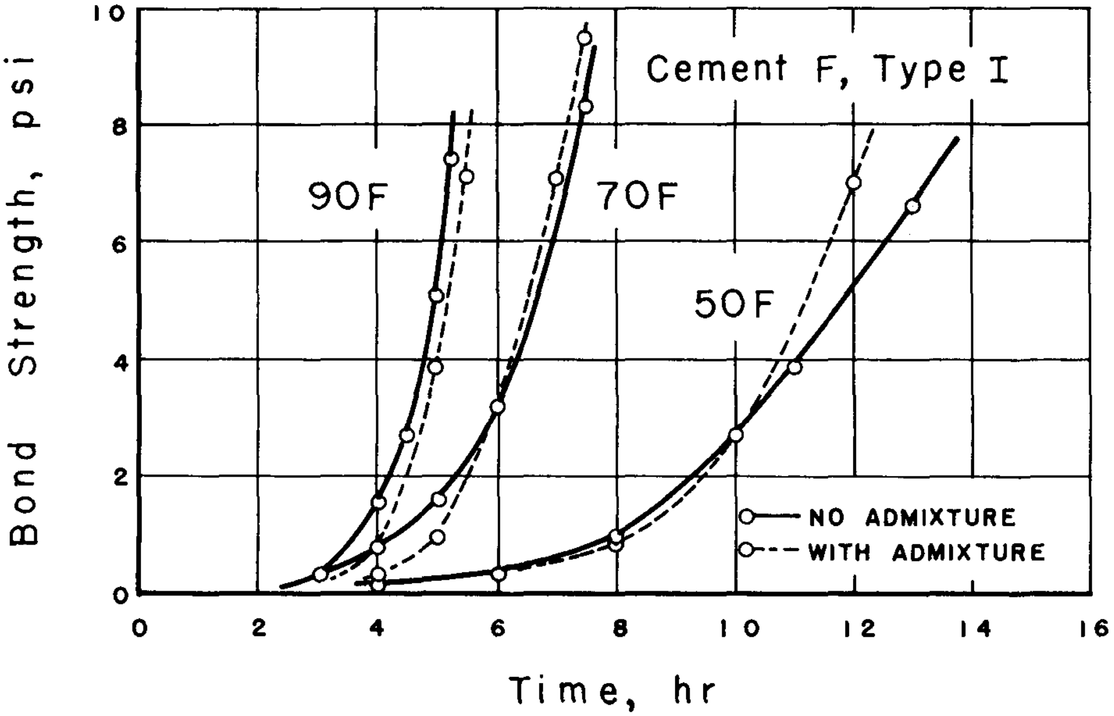

FIG. 7—Effect of water-reducer on rate of hardening.

and of a mixture containing a water-reducing admixture. The mixtures differed only in that the cement factors of the reference and admixture-containing concretes were 5.5 and 4.7 sacks per yard, respectively. Rates of hardening of the two types of concrete were very similar at 70 and 90 F and during the early stages of hardening at 50 F. In the later stages of hardening at this temperature the water-reducing admixture accelerated the hardening rate. The acceleration started at about 10 hr and appeared to increase at a uniform rate, reaching a maximum of about 1½ hr when tests were discontinued.

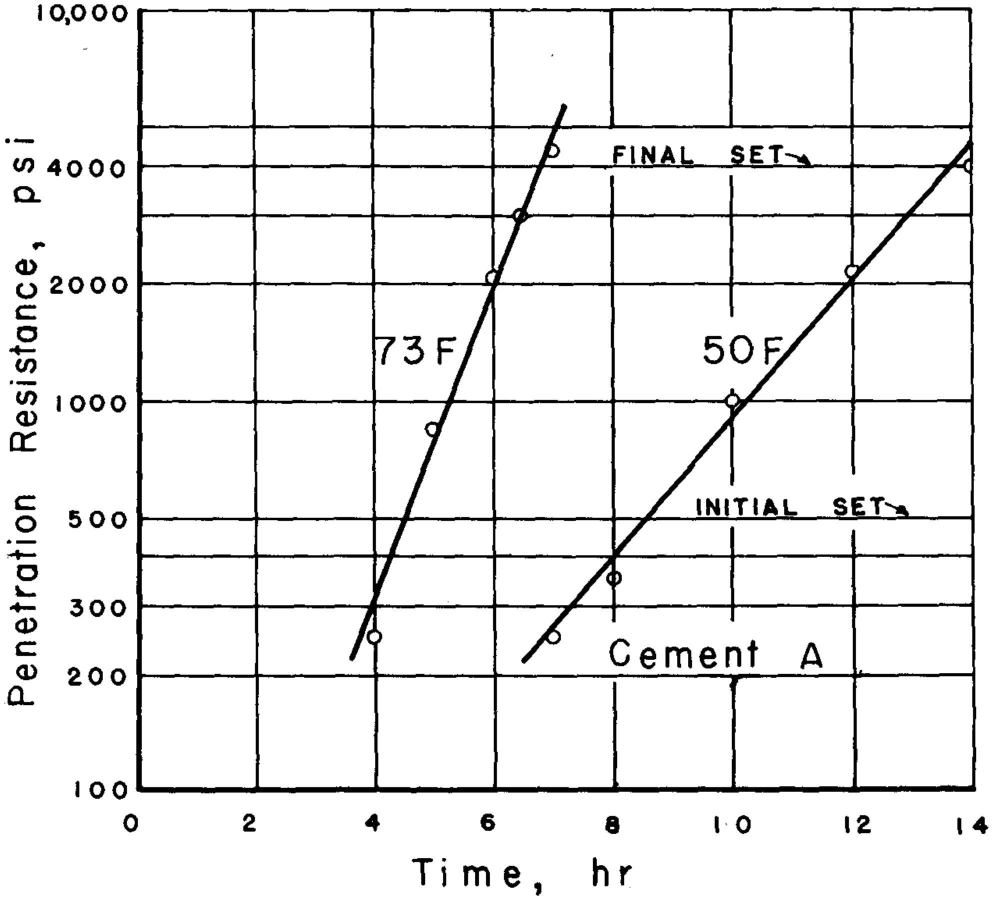

FIG. 8—Effect of temperature on rate of hardening.

EFFECT OF VARIABLES

The results of setting time tests are influenced by several variables. Variations in temperature have appreciable effects on setting times as illustrated in Figs. 1 and 5. In general, the change in setting time per degree change in temperature increases with decreasing temperature. Setting times of concrete vary with type and brand of cement. As shown in Figs. 4 and 6, differences in setting times resulting from a change in brand of Type I cement can be as great as those resulting from a substantial variation in ambient temperature. Tuthill, Adams, and Hemme [*7*] concluded that the time to the vibration limit and the time rate of setting may be influenced more by the brand and possibly the type of cement than by differences in the variety of water-reducing retarders investigated. Vollick [*8*], in Fig. 7 of his paper on water-reducing, set-retarding admixtures, illustrates substantial differences in times of setting of concretes that are similar in all respects except for type of cement. His work indicates that Type III and Type II cements accelerate and retard, respectively, as compared with Type I cement. Mixture proportions, cement factor, slump and, of course, additions and admixtures all influence setting time to some degree. In general, setting times are accelerated by an increase in cement factor and retarded by an increase in slump. In regard to admixtures, recent studies have indicated that time of addition of set-retarding admixtures may have an effect on time of setting of the concrete, the retardation usually increasing with delay in introduction of the admixture [*9,10*]. However, the effect varies with the nature of the cement and the admixture.

REPORTING RESULTS

In a recent publication Polivka and Klein [*11*] and Loughborough [*12*] plotted rate of hardening test data on a semilogarithmic scale, demonstrating that the resulting curves are approximately straight lines. The ASTM subcommittee on setting time is presently considering the desirability of specifying in Method C 403 that rate of hardening test data be plotted on a semilogarithmic scale. In well-controlled tests, data points so plotted generally do not deviate appreciably from a straight line, whereas a linear plot yields a curved line. Some,

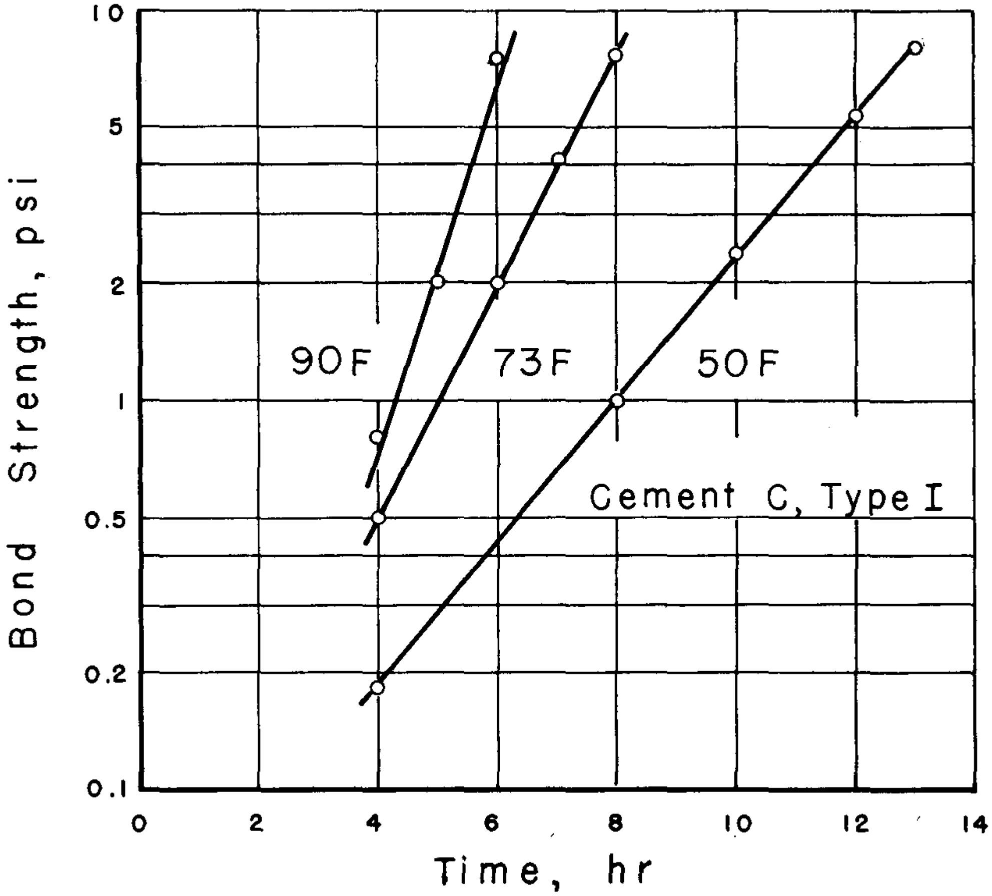

FIG. 9—Effect of temperature on rate of hardening.

who prefer the semilogarithmic scale to the linear scale, maintain that an average straight line can be drawn more accurately than an average curve through the plotted points derived from the required three separate rate of hardening tests. They also point out that the slope of the curve obtained by a linear plot is undergoing the most rapid rate of change in the critical area of initial set, and unless an adequate number of determinations are made at this stage of hardening the estimation of initial setting time may be inaccurate. Others, who prefer the linear plot, feel that it provides a more meaningful picture of the manner in which the concrete is hardening. They also feel that, since the data points on a semilogarithmic plot seldom form a perfectly straight line, the position of the line is a matter of judgment on the part of the operator, while with a linear plot a smooth curve can usually be fitted fairly closely to all data points. From data available, it appears that the setting times determined by either method of plotting are essentially the same. Figure 8 is a semilogarithmic plot of the penetration data plotted linearly in Fig. 1. Figure 9 is a semilogarithmic plot of the bond pullout data plotted linearly in Fig. 5. Data from both test methods yield approximately straight lines when the penetration resistance and bond stress are plotted on the logarithmic scale.

REPEATABILITY

The repeatability of rate of hardening determinations made in accordance with ASTM Method C 403 has not been positively established. Preliminary investigations by members of Subcommittee III-n, ASTM Committee C-9, indicate that results are repeatable to within plus or minus 15 min, under well-controlled testing conditions. A similar degree of repeatability is indicated for the bond pullout pin method. It is anticipated that the repeatability of ASTM Method C 403 will be improved by further refinement of testing procedures as additional experience is gained in use of the method. For example, there are indications that penetration resistance values may differ substantially, depending upon the size of needle being used, in which case it may be desirable to specify the sizes of the needles to be used over various ranges of penetration resistance. The influence of temperature variation from center to edge of specimen and the effect of boundary restraint also need further elucidation.

APPLICATION OF SETTING TIME TESTS

In Acceptance Specifications:

A suitable method for measuring the rate of hardening and determining the setting time of concrete was essential to the development of ASTM Specification for Chemical Admixtures for Concrete (C 494 – 65 T). This specification set tolerance limits for effect on setting time of five types of chemical admixtures as determined by the penetration resistance method. The tolerances were established on the basis that the temperature of each of the ingredients of the concrete mixtures, just prior to mixing, and the temperature at which the time of setting specimens are stored during the test period shall be 73 ± 3 F. A retarding admixture must reach initial set at least 1 hr later but not more than 3 hr later than the reference concrete and must reach final set not more than 3 hr later than the reference concrete. An accelerating admixture must reach initial set at least 1 hr sooner but not more than 3 hr sooner than the reference concrete and must reach final set at least 1 hr sooner than the reference concrete. An admixture that is required neither to accelerate nor retard must reach both initial and final set not more than 1 hr sooner or 1 hr later than the reference concrete. The purpose of Specification C 494 is to provide methods and criteria for evaluating chemical admixtures under highly standardized testing conditions in the laboratory and are not intended to simulate actual job conditions.

In Field Concreting:

Job conditions frequently require a greater degree of and closer control of retardation or acceleration than is required under ASTM Specification C 494. The more stringent requirements may result from unusual temperature or exposure conditions at time of concrete placement; structural considerations, such as avoiding cracking of the concrete deck when steel bridge girders deflect during placement of the deck; avoidance of cold joints in mass concrete; or special types of concreting operations, such as slip form construction. By prior studies in the laboratory under simulated job conditions, utilizing the concreting materials to be used in the work, time of setting tests are invaluable in planning concreting operations and determining whether special measures will be required for set control. If control is required, the type and dosage of admixture can be tentatively selected, subject to verification and adjustment based on time of setting tests made on the job during actual construction.

Precise correlations between time of setting test results and slab finishing operations have not been reported in any published work. It seems reasonable to believe, however, that setting time tests made on slab concrete at the jobsite would provide guidance in scheduling finishing operations.

Both the penetration resistance method and the bond pullout pin method have been used advantageously in the field. Because it is a tentative standard test method and utilizes less cumbersome apparatus, the former method has been used more extensively. However, an engineer for one construction agency[3] utilizes the latter method stating: "Although the pin pullout apparatus is quite bulky and awkward for general field use, it is advantageous in that the rate of hardening test is made on actual job concrete." In this instance the method was being used in the field on three large highway projects to determine the influence of different retarders on rate of hardening of pavement concrete under field conditions.

Determinations of setting times made on small specimens may not be directly applicable to the same concrete in a structure. It has been noted by one construction engineer[3] that concrete in the structure sets up at a slightly faster rate than concrete in beam specimens used for the bond pin pullout test, probably due to the differences in the concrete volume and resultant temperature rise.

Wallace and Ore [*13*] reported that tests performed on samples of concrete at the batch plant do not reflect the influence of the increasing temperature occurring within massive blocks in the dam. They conducted tests in which mortar samples for penetration resistance tests, sieved from the mass concrete at a temperature of about 52 F, were immediately taken into each of three rooms wherein the air temperature was maintained at 50, 75 and 100 F while rate of hardening tests were performed. The influence of predictable temperature cycles for various job conditions on the rate of hardening of mass concrete could be estimated from the data thus obtained.

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[3] Peter Smith, Sr., materials engineer, Ontario Department of Highways, private communication.

Difference in conditions of exposure between the time of setting specimens and the concrete in a structure can lead to misapplication of time of setting test results. This is particularly true in the case of flat slab work where the time of setting of the slab may be strongly influenced by wind velocity, effect of the subgrade, and the radiant heat of the sun as pointed out by Schutz [*14*].

SUMMARY

The development of methods of test for measuring the rate of hardening of concrete, on the basis of which setting times can be defined and determined, has been an important contribution to concrete technology. Factors that affect the setting time of concrete can now be evaluated with reasonable accuracy and concreting operations planned or adjusted accordingly. By means of time of setting tests the need for set-controlling admixtures can be established with certainty and type and dosage can be determined on a quantitative basis. In interpreting results of setting time tests for application to an actual concreting operation, however, one should give due consideration to disparities between the mortar or concrete in the test specimens and the concrete in the structure, such as relative surface to volume ratios, exposure to wind and sun, or exchange of heat with the subgrade.

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STP169A-EB/Apr. 1966

Freshly Mixed Concrete

AIR CONTENT AND UNIT WEIGHT

By F. F. Bartel,[1] Personal Member, ASTM

Tests for air content and unit weight of freshly mixed concrete are made frequently both in the field and in the laboratory. Procedures are outlined in various ASTM standards which are straightforward and well within the capabilities for performance and understanding of the average concrete technician. Further, a minimum of training is required before proficiency in the testing technique is acquired. Nevertheless, an understanding of the limitations of the various methods, together with an appreciation for carefully following details of the procedures, is essential to obtaining and interpreting results.

Tests for air content and unit weight are made of fresh concrete to provide a control on these properties in the hardened concrete and also to determine volume of concrete being produced from a given batch and its unit cement content. The significance of air content and unit weight in hardened concrete is discussed in detail in this publication by Helms.[2] Air content most commonly is determined to insure the presence of small amounts of air prescribed in concrete in order to obtain its beneficial effects on resistance to freezing and thawing and effects of deicing agents applied to concrete for snow and ice removal. Tests for unit weight are made to control weight *per se* of both lightweight and high-density concretes. For all concrete, unit weight determinations are used to establish the volume of batch produced and to establish actual cement contents.

The interrelation between air content and unit weight should be obvious; an increase in volume of air results in a lower unit weight, whereas a reduction in air content increases unit weight. Therefore, testing procedures for these properties are combined in ASTM methods and should be performed in sequence in the field.

Test Methods

Three tests for air content of fresh concrete, using gravimetric, pressure, or volumetric methods, currently are standards of the ASTM. Each of the procedures has its advantages and disadvantages, and it is expected that the method most appropriate for the conditions and materials will be used. Tests for unit weight are part of the gravimetric method.

Gravimetric Method:

In the gravimetric procedure the unit weight of concrete is determined in accordance with ASTM Test for Weight Per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138). The test is made of a sample of concrete in a measure of specified volumetric capacity. Concrete containing aggregate graded up to 2 in. may be tested in a $\frac{1}{2}$-ft^3 container; where aggregate up to 3 in. in size is used,

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[1] Civil engineer, Tews Lime & Cement Co., Milwaukee, Wis.

[2] See p. 309.

the 1-ft^3 measure should be employed. A 5-ft^3 container should be used for concrete containing still larger aggregate. The measure is filled in three layers, and each layer of concrete is consolidated by rodding and tapping the sides of the measure a specified number of times. The strikeoff of the top surface must be done "... with a flat cover plate using great care to leave the measure just level full." From the known volume of the measure and the weight of its contents, the weight per cubic foot of concrete can be computed. In Method C 138 details of the calculations are outlined to determine: (*a*) weight per cubic foot, (*b*) volume of concrete produced per batch, (*c*) yield (or volume) of concrete produced per bag of cement, (*d*) relative yield, or ratio of actual volume produced to the designed volume of the batch, (*e*) actual cement factor as number of bags of cement per cubic yard of concrete, and (*f*) air content of concrete.

When the gravimetric procedure for determining air content of fresh concrete is used, the unit weight of concrete as determined is compared with the theoretical unit weight of air-free concrete. The theoretical unit weight is calculated from the weight and bulk specific gravity of each ingredient in the concrete mixture following the calculations outlined in C 138. The gravimetric method for determining air content has serious limitations as a field test. One disadvantage is that an accurate knowledge of batch proportions, specific gravities, and moisture contents of aggregates is essential. For example, an error of two per cent in the moisture content of sand in an average concrete mix, in which a constant amount of water is added at the mixer, will result in an error of one percentile in computed air content. In the field where, in many cases, average moisture contents of aggregates are assumed and consistency is controlled by varying the amount of water added at the mixer to maintain slump, the gravimetric method should not be relied upon for accurate results. Also, an error of 0.02 in the specific gravity of the aggregates will result in an error of about one-half percentile in air content computed under this method. Thus, where lightweight aggregates are used, whose specific gravity is exceedingly difficult to determine, it is not recommended that the gravimetric procedure be used.

In the laboratory, however, where specific gravities and moisture contents are accurately known, reliable results can be obtained for concrete containing normal weight aggregates. Furthermore, the concrete used in the test need not be discarded, as in other procedures; it can be used for other tests or in the preparation of specimens.

Pressure Method:

The pressure method for determining air content of fresh concrete is based on the fact that, in most concrete mixes, the only compressible ingredient is the air entrained or entrapped in the mixture. Klein and Walker [*1*][3] applied Boyle's law, involving the relationship of pressure and volume of gases, to the determination of air content of fresh concrete. Menzel [*2*] refined the apparatus and proposed a standard test procedure.

In the apparatus described in ASTM Test for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231), a predetermined pressure is applied to a column of water above a sample of concrete in a container of known volume. When the proper pressure is applied, the drop in water level in the neck of the calibrated apparatus indicates the air content of the concrete directly. The instrument must be calibrated for use at various localities if differences in altitude

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[3] The italic numbers in brackets refer to the list of references appended to this paper.

are considerable. Also, the results vary with changes of barometric pressure. A complicating feature of the test is that the pressure may compress air within the interstices of nonsaturated, porous aggregates, and a higher than true air content of concrete may thus be indicated. This complication may be compensated for by determining a correction factor for the air within the aggregate as outlined in Method C 231. In the case of slag vesicular volcanic aggregates or lightweight aggregates, however, this aggregate correction factor may be quite large and difficult to determine accurately. The ASTM method provides for three sizes of containers, 0.20, 0.40, and 2.50 ft^3, for testing concrete having aggregate of 2, 3, and 6-in. max size, respectively.

The major advantage of the pressure method is that no knowledge of specific gravities, moisture contents, or batch quantities of the concrete mixture need be available to determine its air content. Possible inaccuracies when porous or nonsaturated aggregates are used have been referred to previously. Samples of concrete tested in this apparatus must, of course, be discarded and cannot be used in specimens or for further testing because of the contact of concrete with water during the test.

A modification of the ASTM standard pressure apparatus has been developed by the Washington State Highway Dept. [*3*]. In this apparatus, a known volume of air at an established pressure is released to contact concrete in a sealed container; the drop in pressure provides a measure of the amount of air within the concrete. The apparatus has the advantage of not having test results influenced by changes in barometric pressure. Compressibility of air within the aggregate must be taken into account in test results as it must be with results from the Klein-Walker apparatus. In the original "Washington" meter no water was used in the testing. With that apparatus it was necessary to use a flat plate in striking off the sample in the test container to achieve maximum accuracy in obtaining an exact volume of concrete; even small errors in striking off the test sample produced large errors in measured air content. The currently available commercial meters of the "Washington" type use small quantities of water (about 4 oz) to fill the space over the sample precisely, thereby greatly reducing inaccuracies resulting from imperfect strikeoff.

The availability of six or more commercially produced air meters operating on the pressure principle but varying in details of design posed a problem to the ASTM subcommittee having immediate jurisdiction over Method C 231. Should a new method be published for each manufacture of pressure meter or should only one apparatus be considered standard? The decision reflected in C 231 was to spell out the essentials of all pressure meters and to describe, in detail, the meter as refined by Menzel [*2*]. For other pressure air meters, the manufacturer's instructions should be followed, and the meter calibrated following the procedure outlined in Method C 231 by using a calibration cylinder of known volume to represent a given volume of air.

Volumetric Method:

Inaccuracies in determining air content of fresh concrete containing porous aggregates by the gravimetric and pressure methods have been described. The volumetric procedure, however, can be used with such materials to obtain accurate measurements of air content. Further, as with the pressure method, no knowledge of specific gravities or moisture contents of ingredients need be known.

With the apparatus described in ASTM Test for Air Content of Freshly

Mixed Concrete by the Volumetric Method (C 173), water is filled to mark over a sample of concrete in a container of known volume. The apparatus is sealed; then concrete and water are intermingled and agitated until the air in the concrete is displaced by washing action. The drop in water level from its original mark provides a direct measure of the air content of the concrete. In the test, mixing of water and concrete is repeated until no further drop in water level indicates the removal of all air from the mixture. The method requires the use of a container for concrete of not less than 0.2 ft^3 when aggregates up to 2 in. are used; however, for routine work, a bowl of not less than 0.075 ft^3 may be used.

The chief disadvantage of the volumetric method is in the physical effort required to agitate water and concrete sufficiently to remove the air. Accordingly, a smaller apparatus is permitted by ASTM C 173 for routine work; the volume of concrete tested is small but not inconsistent with the findings of Pearson and Helms [*4*] on the effect of sampling errors in air-content tests.

Other volumetric procedures, such as the rolling method developed by Menzel [*2*], the hook-gage or "Indiana Method" described by Miesenhelder [*5*], and the "Ohio Method" [*6*] using a hook gage, test larger samples of concrete. With Menzel's volumetric apparatus the quantity of water added to compensate for air removed from a known volume of concrete by mixing with water (on a rolling apparatus) provides a measure of the air content. In the "Indiana Method," air is removed from a known weight of concrete by stirring in water; the air content is computed from the unit weight of air-free concrete determined by displacement in the test, and the unit weight measured prior to removal of the air. Miesenhelder [*5*] reported the disadvantages of the latter method to be the difficulty in maintaining the necessary accuracy of scales under field conditions and the possibility of incomplete removal of air because of the physical exertion required in the stirring process.

A device known as the Chace Air Indicator has been used extensively to measure air content of fresh concrete. It involves a volumetric procedure utilizing a pocket-sized graduated glass tube into which is fitted a brass cup 1 in. in diameter by $\frac{1}{2}$ in. high attached to a rubber stopper. A sample of mortar is secured from the concrete, consolidated in the brass cup, and struck off to make the cup level full. The graduated glass tube is filled with isopropyl alcohol, and the brass cup inserted into it. After adjustment of the alcohol level to mark, the tube is inverted from horizontal to vertical several times until all mortar has been displaced out of the cup into the alcohol. In the course of this intermingling of mortar and alcohol, the air is washed from the mortar. The drop in level of the alcohol from its original mark is a measure of the air content of the mortar; calculations dependent on the volume of mortar in the concrete yield an indicated air content of concrete. Tests reported by Willetts and Kennedy [*7*] under laboratory conditions showed the Chace Indicator to be accurate within plus or minus 0.6 percentile two thirds of the time for air-entrained concrete. Additional studies of the air indicator were reported by Grieb [*8*] and Newlon [*9*]. Its principal use would appear to be as a speedy screening device to indicate when air contents apparently are approaching or do not conform with specification limits. When this is indicated, the appropriate ASTM method of test for air content should be used for acceptance or rejection of the concrete.

COMPARISON OF TEST RESULTS

The existence of three ASTM procedures for determining air content of fresh concrete logically raises the question as to which is most accurate. Many studies have been made on this subject. In the ASTM *Symposium on Measurement of Entrained Air in Concrete* [*10*] the results presented by the various authors are in substantial agreement. Results of gravimetric tests agreed with other procedures as long as specific gravities and moisture contents of ingredients were known, pressure method test results were in accord with other data when concrete was made with dense aggregates, and volumetric procedures gave substantially the same air contents as did the other methods as long as all of the air was removed from the sample tested. Tests by Britton [*11*] using the current ASTM gravimetric, pressure, and volumetric procedures gave comparable results, aside from the limitations mentioned above, for tests carefully conducted.

SIGNIFICANCE OF TEST RESULTS

Tests for air content of fresh concrete are most commonly made in the field on concrete that is assumed to contain entrained air. Air entrainment is obtained by using an air-entraining cement or by adding an air-entraining admixture to concrete or by using an air-entrained admixture to supplement the air entrainment achieved by use of an air-entrainment cement. Numerous tests have demonstrated that purposefully entrained air increases the resistance of concrete to destructive agencies such as freezing and thawing, sea water, and salts used for removal of snow and ice from pavements. Along with these beneficial effects, unfortunately, there is usually a decrease in the strength of concrete, especially in relatively rich mixes. Thus, the problem in the case of air-entrained concrete is to control the air content to ensure the beneficial effects without unduly reducing its strength.

Studies by Wuerpel [*12*], Gonnerman [*13*], and others show the need for a minimum of about three per cent entrained air in concrete mixtures containing 2-in.-max size aggregate to secure the full benefits of increased resistance to freezing and thawing. On the other hand, tests by the Portland Cement Assn. [*13*] and the National Sand and Gravel Assn. [*14*] indicate that, *for a given water-cement ratio*, each percentile of entrained air reduces compressive strength about five per cent. This reduction in strength, however, can be counteracted by reproportioning the mix by reducing water and sand contents to maintain the slump and volume of mortar in the concrete. Thus, by reproportioning of the mix, it is possible that the strength of lean, air-entrained concrete mixtures (containing up to about five bags of cement per cubic yard) actually may be greater than that of normal concrete. In the case of rich mixtures, the reduction in strength may be only 10 to 15 per cent instead of the 20 to 25 per cent which could be expected were the mix not reproportioned. It is from this background of information, together with field experience, that most specifications require four to seven per cent air in freshly mixed air-entrained concrete.

There are exceptions, however, where the 4 to 7 per cent limits on air content should not be applied. In mass concrete, for example, in which cobbles of aggregate 6 in. or more in diameter are used, these limits may be applicable to that portion of the concrete which would pass a 2 or 3-in. sieve, but they would be too high for the concrete as a whole. As a matter of fact, the Corps of Engineers [*15*] wet screens its concrete containing a nominal maximum size aggregate of 3 to 6 in. through a $1\frac{1}{2}$ or 3-in. sieve before testing for air content. The Bureau of Reclamation generally requires removal of larger sizes of coarse aggregate by handpicking before a test for air content is made. In concrete mixtures used in Kansas pavements in which the maximum size of aggregate is about $\frac{3}{8}$ in., the 4 to 7 per cent limits on air content should probably be raised.

The above discussion of exceptions to the usual specification limitations on air content indicates that the air content of the mortar or cement paste portion of the concrete should be of principal concern. Wuerpel [*16*] has pointed out that the optimum air content for the mortar phase of concrete should be 8.5 to 9.5 per cent. Powers [*17*] and Klieger [*18*] have shown that the air content of fresh concrete should be governed by the spacing and size of voids in the cement paste. In concrete of a given air content, numerous small air bubbles closely spaced throughout the mass are more effective in securing the beneficial effects of air entrainment than are a smaller number of relatively large voids.

The air content of concrete containing a fixed amount of air-entraining admixture added at the mixer, or as an addition interground with the cement, varies with many factors. It is important that the effects of these factors be understood so that test results obtained may be evaluated and adjustments made to secure compliance with specifications. Less air will be entrained in rich mixtures than in lean ones under given conditions. Concrete of high slump will ordinarily entrain more air than that of low slump. The temperature of the concrete is important in that more air is entrained by the same dosage of air-entraining admixture at 70 F than at 100 F, and more at 40 F than at 70 F [*19*,*20*]. The quantity of air will increase with mixing up to a point and then will gradually decrease; in transit-mixed ready-mixed concrete, for example, tests have shown air content to increase during about the first 12 min of mixing and then slowly to decrease [*21*]. The grading of the aggregate, particularly that of the sand, also has its effect on air content; Walker and Bloem [*19*] found that the air content of concrete increased as the proportion of sand in the Nos. 30 to 50 size increased.

So far discussion has been confined to air content of concrete as mixed. However, handling and placing of concrete on the job can affect the air content of concrete *in place*. And, of course, it is the air voids in the hardened concrete that secure the beneficial effects attributed to air-entrained concrete. There are indications that pumping of concrete may remove some air from certain mixtures. Repeated handling and agitation of concrete prior to placing might cause loss of air content. Vibration in consolidating concrete in the forms may also remove air from the mass [*22*]. It should not be overlooked that the air in the lower portion of a deep section will be compressed by the weight of concrete above it; this will affect the volume of air but probably not the spacing, number of air voids, or the durability of the concrete.

Nevertheless, even with all these complications, tests of fresh concrete for air content by the ASTM methods described here constitute the best means for controlling the end result in the field. These test methods, coupled with intelligently prepared specification limits, should ensure the desirable properties of air-entrained concrete.

Air content determinations are sometimes made to ensure that nonair-entrained concrete is "normal"—that it does not contain more than about 1 to 1.5 per cent of air. This is particularly necessary for mixtures for heavy-duty floors or for very high-strength concrete which might be used in prestressed designs or heavy-load-carrying columns. Many manufacturers of metallic aggregates for heavy-duty floors, for example, recommend that their products not be used with air-entrained concrete. It is difficult to produce high compressive strengths (5000 psi, for example) with air-entrained concrete, particularly in hot summer temperatures. This makes apparent the desirability of making an air-content test, at the time high-strength concrete is placed, to ensure that even a moderate amount of air is not being entrained.

Unit weight tests of fresh concrete, as previously stated, are used to control weight *per se* of both lightweight and high-density concretes and to establish the size of batch (or yield) being produced. In the case of lightweight aggregates, it is extremely difficult to determine accurately specific gravity and absorption. For a given aggregate, the density of individual particles varies with size, with the larger particles being less dense because they have been expanded more. Thus, as gradation of the lightweight aggregate changes even a relatively small amount, the specific gravity will change significantly. Further, it is impractical to bring these aggregates to an exact condition of saturated-surface-dry, especially when particle shape is rough and irregular.

This difficulty in accurately determining specific gravity and absorption of lightweight aggregates makes conventional concrete mix design procedures inaccurate. The unit weight procedure for proportioning and controlling concrete mixtures made with lightweight aggregates, however, circumvents these difficulties. Details of the procedure are outlined in the Recommended Practice for Selecting Proportions for Structural Lightweight Concrete of the American Concrete Inst. [*23*]. The weight of all materials to be used to produce a cubic foot of concrete is estimated on the basis of experience, from data supplied by the aggregate manufacturer, or from weights per cubic foot of dry loose aggregate. On the basis of the estimate, a trial mix of concrete is mixed and tested. If the actual unit weight of the fresh concrete differs from the estimate appreciably, it will be necessary to increase or decrease quantities of materials until the yield approaches 27 ft^3 to the cubic yard.

The unit weight of high-density concrete has been of interest for many years where it was used in such things as counterweights, dam gates, and so on. Recently the need for radiation shielding in various nuclear devices has increased the use of high-density concrete. In general, the greater the density of a material, the greater the ability to shield against radiation. Concrete, with its relatively high-weight to low-cost ratio, has been used extensively for this purpose. Where space limitations prevail, special high-density aggregates have been used to increase unit weight of concrete. The subject of radiation effects and shielding is discussed in detail in this publication by Polivka.[4] Obviously, it is the weight of hardened (not fresh) concrete which is of concern for radiation shielding; however, the control must be exercised through unit weight determinations of freshly mixed concrete.

The use of Method C 138 to determine yield of concrete has been referred to previously. Yield as such is defined as the volume of concrete produced per bag of cement. This yield unit is a holdover from times when most cement was batched volumetrically by the bag; principal interest was in knowing how much concrete was being produced for each bag of cement. Today more attention is paid to the term "relative yield" which was added to Method C 138 in 1963. This is defined as the ratio of actual volume of concrete obtained to the volume as de-

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[4] See p. 298.

signed for the batch. Relative yield is of special importance to the commercial transactions between a ready-mixed concrete producer and his customer. It indicates whether a full cubic yard is being delivered or whether the batch is short of its design volume.

SUMMARY

Tests for air content and unit weight of fresh concrete, carefully made in accordance with the appropriate ASTM test method, will yield an accurate measurement of the amount of air, weight, and volume of concrete being produced. Tests for air content, coupled with intelligently selected specification limits, can ensure the beneficial effects of entrained air in hardened concrete. Unit weight tests are reliable for controlling yield of concrete, and for evaluating unit weight when stated in specifications.

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Hardened Concrete

PETROGRAPHIC EXAMINATION

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Petrography is the scientific description of the formation and composition of rock, including the systematic classification of rocks. Petrographic examination of hardened concrete—a man-made rock—is the examination of concrete by the techniques used in petrography, to determine the formation and composition of the concrete and to classify it as to its type, condition, and serviceability.

This paper is intended to show that petrographic examination provides information useful in evaluating hardened concrete, to note the problems inherent in its use, to outline what it involves, to describe the kinds of information that it can produce, and to show how this information can be applied.

The questions that materials testing and evaluation tries to answer are: (*a*) "How will this material behave in use?" or (*b*) "Why did this material behave in use in the way it did?" The first question is never unequivocally answered except after-the-fact, by determining how the material did behave in use. The most useful method for developing practical information upon which to make decisions that depend on prediction of probable behavior of materials is the study of why materials behaved in use as they did.

Testers of materials are unable to compress time or to anticipate and reproduce the environment that the material will experience. Generally they use standardized procedures not directly related to the specific environment or that do not determine the particular properties relevant to performance in the specific instance. Thus, testing construction materials amounts to obtaining certain kinds of information about certain samples in specified conditions and extrapolating to the conditions of intended use insofar as they can be predicted.

Petrographic examination of hardened concrete is included among the subjects in this volume because it helps to improve the extrapolation from test results to performance in use. It offers direct observational information on what is being tested and what is in the structure, giving another way of appraising the relation between samples and materials in use and judging how similar the two are.

COMMUNICATION PROBLEMS

A petrographic examination of concrete ordinarily begins and ends with a problem of communication between the person who requests the examination (usually an engineer) and the person who makes it (usually a petrographer). Unless the two succeed in producing a clear, mutually understood statement of the problem, they cannot expect a clear, useful answer to be economically obtained. The engineer who asks for an

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examination of a particular concrete suspects that the concrete is unusual; the more clearly he defines the features prompting his interest, the more he directs the petrographer toward the important aspects. The engineer may not be familiar with the techniques that the petrographer may use or with his approach; the petrographer may not realize the engineer's responsibility for decision and action, may not find out all the engineer could tell him about the concrete, and may not realize which petrographic findings are useful and relevant. The petrographer should not expect petrographic results to be taken on faith; the rationality of the techniques producing them should be demonstrable. Both should remember that the essentials of petrographic examination of concrete are practiced anytime anyone looks intelligently at concrete either in a structure or as a specimen and tries to relate what he can see to the past or future performance of the concrete. On this basis, it is clear that many most useful petrographic examinations are made by inspectors, engineers, chemists, physicists—anyone concerned with the production or use of concrete. No one should hesitate to examine concrete with all available means; all, from novice to expert, should question the indicated conclusions and verify them in as many ways as possible.

METHODS

A recommended practice for petrographic examination of concrete aggregates has been standardized [*1*].[2] Techniques of petrographic examination of hardened concrete have been described and discussed [*2–25*]. They have not been standardized for several reasons including: the considerable variety of techniques that are used in different laboratories, the wide variety of purposes for which examinations are made, the rapidly increasing uses in the recent past of a variety of instruments, and kinds of data clearly applicable in petrographic examination of cement paste and hardened concrete but not yet available in many laboratories in which petrographic examinations of concrete are made.

At present I know of no laboratory where petrographic examinations of concrete are made that is equipped to use all the methods that have yielded useful information and no one person who has digested the available approaches and developed the ability to choose the particular combination of techniques best suited to each problem encountered. Concretes are more complex than most rocks used as aggregates; their constituents are less well known; concretes change through time more rapidly than most aggregates. All of these circumstances combine to make each petrographic examination of concrete unique and thus to make the methods harder to generalize and standardize. Each examination presents some new facet for the petrographer who is willing to learn.

Between 1950 and 1955 remarkably rapid advances in knowledge of the calcium silicate hydrates [*26–28*] were brought about in part by work sponsored by the Building Research Station. J. D. Bernal and his students, then working at Birkbeck College, and others in the United Kingdom, proceeded by synthesis of compounds and study of naturally occurring minerals, making use of X-ray crystallographic methods, optical methods, and static dehydration. The interest thus aroused among mineralogists and chemists, especially in the United Kingdom and the Soviet Union, has led to a greatly increased knowledge of natural and synthetic calcium silicate hydrates [*29*], recently summarized by Taylor [*30, 31*] and from another approach by Belov and co-workers [*32*].

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

In 1955 differential thermal analysis had been usefully applied [*24,25*] but not widely investigated. The use of electron microscopy in the study of portland cement hydrates had not advanced very far in the United States, and differences of opinion on proper specimen preparation and ambiguities in interpretation made it difficult for persons not expert in the field to assess the value and meaning of the results. The potentialities of these methods and others for characterizing calcium silicate hydrates, calcium aluminate hydrates, and other phases in hydrated cement paste could be recognized, but most laboratories where petrographic examinations of hardened concrete were made were not equipped with instruments more sophisticated than the polarizing microscope and were not yet prepared to relate and to combine the information available from visual observation and light microscopy with that available from X-ray diffraction, differential thermal analysis, and electron microscopy and diffraction.

These newer techniques and the results they yield offered an opportunity to understand in much more detail than previously had been possible the chemical reactions that had gone on in a concrete being examined, because they suggested that it would soon be possible to characterize the hydration and reaction products more clearly and thus to reconstruct in detail the chemical reactions of normal hydration or abnormal deterioration or both in the concrete, and to evaluate, with much more certainty than is possible without fairly complete identification of the reaction products, the relative roles of chemical attack and physical attack in producing deterioration. The goal of relating better established and more familiar techniques in petrographic examination of hardened concrete to the more intimate and detailed insights made possible by the newer techniques remained to be achieved, and while progress since 1955 is considerable, the task is not complete.

The term, petrographic examination of hardened concrete, does not imply that a microscope or any other particular instrument necessarily is used; it does imply examination to discover what recognizable constituents are present and how they are arranged in space. Often it implies an evaluation of quality and condition of the concrete—an evaluation sometimes based on quantitative information.

ASTM Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (C 116 – 65 T) and Test for Compressive Strength of Molded Concrete Cylinders (C 39) requiring observation of "type of failure and appearance of the concrete" and "type of fracture if other than the usual cone," respectively, implicitly require partial petrographic examination—the part dealing with defects and departures from the norm—but offer no instructions on how to conduct it. Several specifications under the jurisdiction of ASTM Committee C 13 on Concrete Pipe include as one basis for rejection "measurements and inspection" to ascertain whether the product conforms to the specification as to design and freedom from defects (Specification for Concrete Sewer, Storm Drain, and Culvert Pipe (C 14), section 35; Specification for Reinforced Concrete Culvert, Storm Drain, and Sewer Pipe (C 76 - 65 T), section 27; Specification for Concrete Pipe for Irrigation or Drainage (C 118 – 65 T), section 20; Specification for Reinforced Concrete Low-Head Pressure Pipe (C 361 – 65 T), section 32; Specification for Concrete Drain Tile C 412), sections 16, 17; Specification for Perforated Concrete Pipe (C 444 – 65 T), sections 13, 19; Specification for Precast Reinforced Concrete Manhole Sections (C 478 – 65 T), sections 24, 27; Specifi-

TABLE 1—OUTLINE FOR EXAMINATION OF CONCRETE WITH EYE AND HAND LENS.

Coarse Aggregate	+ Fine Aggregate	+ Matrix	+ Air	+ Steel
CONSTITUENTS				
Maximum dimension, in inches, in the range——>d>——[a] *Type:* 1. Gravel 2. Crushed stone 3. Mixed 1 and 2 4. Other (name) 5. Mixed 1 +/or 2 +/or 4 If 1, 2, or 4, homogeneous or heterogeneous	type: 1. natural sand 2. manufactured sand 3. mixed 4. other 5. mixed 1 +/or 2 +/or 4 if 1, 2, or 4, homogeneous or heterogeneous	color, by comparison with National Research Council Rock Color Chart (1948) *color distribution:* 1. mottled 2. even 3. gradational changes	more than 3 per cent of total, predominantly in spherical voids less than 3 per cent of total, abundant nonspherical voids color differences between voids and mortar?	type, size, location
Lithologic types Coarse aggregate more than 20, 30, 40, 50 per cent of total			voids empty, filled, lined, or partly filled	
FABRIC				
Shape Distribution Packing Grading (even, uneven, excess, or deficiency of size or sizes) Parallelism of flat sides or long axes of exposed sections, normal to direction of placement +/or parallel to formed and finished surfaces[b]	distribution particle shape grading preferred orientation } as perceptible	distribution	shape distribution grading (as perceptible) parallelism of long axes of irregular voids or sheets of voids: with each other; with flat sides or long axes of coarse aggregate	voids below horizontal or low-angle reinforcement
CONDITION				
Does it ring when hit lightly with a hammer or does it give a dull, flat sound? Cracks? How distributed? Crack fillings? With cores or sawed specimens: did aggregate tear out in drilling or sawing?				rusty or clean? put in rusty or corroded later?

[a] A substantial portion of the coarse aggregate has maximum dimensions in the range shown as measured on sawed or broken surfaces.

[b] Sections sawed or drilled close to and parallel to formed surfaces appear to show local turbulence as a result of spading or rodding close to the form. Sections sawed in the plane of bedding (normal to the direction of placement) are likely to have inconspicuous orientation. Sections broken normal to placement in conventionally placed concrete with normal bond tend to have aggregate knobs abundant on the bottom of the upper piece as cast and sockets abundant on the top of the lower piece as cast.

TABLE 2—OUTLINE FOR EXAMINATION OF CONCRETE WITH A STEREOMICROSCOPE.

Coarse Aggregate	Fine Aggregate	Matrix	Voids
Lithologic types and mineralogy as perceptible Surface texture Within the piece: grain shape grain size—extreme range observed, mm median within range —— to —— mm textureless (too fine to resolve) uniform or variable within the piece From piece to piece: intergranular bond porosity and absorption[a] If concrete breaks through aggregate, through how much of what kind? If boundary voids, along what kind of aggregate? All? All of one kind? More than 50 per cent of one kind? Several kinds?	lithologic types and mineralogy as perceptible shape surface texture grading distribution	color fracture around or through aggregate contact of matrix with aggregate: close, no opening visible on sawed or broken surface; aggregate not dislodged with fingers or probe; boundary openings frequent, common, rare width empty filled cracks present, absent, result of specimen preparation, preceding specimen preparation fly ash[b]	grading proportion of spherical to nonspherical nonspherical, ellipsoidal, irregular, disk-shaped color change from interior surface to matrix interior surface luster like rest of matrix, dull, shining linings in voids absent, rare, common, in most, complete, partial, colorless, colored, silky tufts, hexagonal tablets, gel, other underside voids or sheets of voids uncommon, small, common, abundant

[a] Pore visible to the naked eye, or at ×——, or sucks in water that is dropped on it.

[b] Dark solid spheres or hollow-centered spheres of glass +/or magnetite recognizable at × 9 on sawed or broken surfaces. Other mineral admixtures with characteristic particles visible at low magnification should be recognizable.

Condition: When it is examined at × 6 to × 10 under good light, the freshly broken surface of a concrete in good physical condition that still retains most of its natural moisture content has a luster that in mineralogical terms is subtranslucent glimmering vitreous.[1] Thin edges of splinters of the paste transmit light; reflections appear to come from many minute points on the surface, and the quality of luster is like that from broken glass but less intense. Concrete in less good physical condition is more opaque on a freshly broken surface, and the luster is dull, vitreous going toward chalky. A properly cured laboratory specimen from a concrete mixture of normal proportions cured 28 days that has shown normal compressive or flexural strength and that is broken with a hammer and examined on a new break within a week of the time that it finished curing should provide an example of concrete in good physical condition.

Under the same conditions of examination, when there is reasonable assurance that the concrete does not contain white portland cement or slag cement, the color of the matrix of concrete in good physical condition is definitely gray or definitely tan, except adjoining old cracks or original surfaces.

[1] E. S. Dana, *Textbook of Mineralogy*, John Wiley & Sons, New York, N. Y., 4th edition, 1932, pp. 273–274; revised by W. E. Ford.

cation for Nonreinforced Concrete Irrigation Pipe with Rubber Gasket Joints (C 505 – 65 T), section 26; Specification for Reinforced Concrete Arch Culvert, Storm Drain, and Sewer Pipe (C 506 – 65 T), sections 26, 29; Specification for Reinforced Concrete Elliptical Culvert, Storm Drain, and Sewer Pipe (C 507 – 63 T), sections 26, 29). These provisions for visual inspection and measurements are also requirements for a partial petrographic examination; a person qualified to make the inspection and measurements needs to be familiar with the products specified, because the instructions provided are not detailed. Tables 1 and 2 give outlines for examining concrete as a material but do not refer to dimensions or to quality of formed or finished surfaces.

PURPOSE AND APPROACH

Purpose:

A petrographic examination attempts to answer two general objective questions: "What is the composition?" and "How is it put together?" The first question refers to the recognizable individual constituents present on the scale at which they are considered. The second question refers to structural fabric, that is, the articulation or packing in space of the component elements making up any sort of external form [*33*] or heterogeneous solid body. Both questions may be answered on any useful scale by choice of technique or techniques of appropriate resolving power. The resolving power needed differs depending on the specific questions to be answered.

Approach:

Step one, in any case, is to define the problem in order to find and ask the right questions, those that need to be answered to solve the problem that caused the examination to be requested. These right questions should be answered insofar as they can be in the context, limited as it will be by money, time, instrumentation, and the state of the art. The best petrographic examination is the one that finds the right questions and answers them with maximum economy in minimum time, with a demonstration clear to all concerned that the right questions were answered with all necessary and no superfluous detail. In practice the approach to the ideal varies depending on the problem, the skill with which the questions are asked, and the skill of the petrographer. One measure of the petrographer's skill is knowing when to stop, either because the problem is adequately solved, or, in some cases, because it has been shown to be insoluble under the circumstances.

FABRIC AND COMPOSITION

Fabric—the packing of component elements in space—is the heterogeneity obvious as one looks at a weathered concrete structure or at a broken or sawed surface of concrete. Fabric includes all of the structural elements, ranging in scale from gross to atomic, and comprises both structure and texture as those terms are used in rock description. The fabric appears on the scale of the lift or course or batch or the structural crack, on the scale of the coarse aggregate, on the scale of the sand grains or the air voids in the mortar or the "microcracks," on the scale of the residual unhydrated cement or the calcium hydroxide crystals, on the scale of the hydrous calcium aluminates and the scale of the poorly crystalline hydrous calcium silicates in the hydrated cement paste, or on the scale of the atomic structure of any crystal forming a part of any of the structural components. Rocks, fabricated metals, and yeast-raised bread are other examples of substances with fabric. The closest naturally occurring analogue among rocks to the fabric of concrete is graywacke conglomerate with abundant matrix. The closest naturally occurring analogue to hardened cement paste is silty clay.

Fabric and composition together define, characterize, and form the basis for descriptive classification of solid multicomponent substances. Composition and fabric are so closely interrelated in concrete that they cannot be clearly separated.

One important decision in a petrographic examination is the decision whether the request for the petrographic examination was made because of problems created by the physical structure of the concrete or because of problems created by its chemical composition. Problems arising from the physical structure or fabric include inadequacies in mixing, consolidation, and inadequacy of the air-void system to provide frost resistance. Problems arising from the chemical composition include those resulting from errors in batching, reactions between cement and aggregate, reactions between a contaminant and cement paste, and reactions between cement paste and solutions from external sources. Were the construction practices employed suitable for producing concrete capable of giving satisfactory service in the particular environment and exposure? Were the materials that were chosen susceptible to participation in chemical reactions that have deleterious consequences? Was there a failure to modify the environment, for instance, by improving the drainage so as to increase the ability of the concrete to survive it? Is the difficulty principally physical or principally chemical? Usually several causes have interacted, but one is probably the originator; if it can be identified, the appropriate techniques are more clearly pointed out.

Investigating composition and fabric provides a specific, unique definition of what is being examined. The standard tests do not always supply information that permits discrimination between one piece of concrete and another, but direct observation on the relevant scale does. There are *n* possible concretes all having 2-in. slumps, with air contents of 5 per cent, with 4500-psi compressive strength at 28 days, but the No. 2 cylinder in the set of 3 broken on Day A in Laboratory B is unique and different, perceptibly and logically, from Nos. 1 and 3 and from all the members of the other possible sets, and its top is different from its bottom as cast. The salient lesson from the study of composition and fabric of concrete is the individuality and uniqueness of each structure or part of a structure, of each specimen and each thin section; this individual combination of fabric and composition reflects the history and conditions the future performance of the concrete. What is investigated at any time is particular concrete, not concrete in general. Each structure and each part of a structure is unique in terms of composition, fabric, history, and exposure.

COMPARISONS

To say that each structure and specimen is unique does not mean that comparisons are useless or impossible; they are essential, and concretes can be rationally grouped and usefully compared within classes and between classes, if the basis for the grouping is objective. Each comparison is an abstraction that leaves out of account some characteristics of the things compared, so that it is necessary to bear in mind that the accidentally or deliberately omitted factors may prove to be important. Paste, mortar, or concrete of known proportions, materials, age, and curing history offer the logical basis for comparison and extrapolation; laboratory specimens made to be examined or salvaged just after having been tested for strength provide a good source of such comparative material. Specimens exposed to laboratory air outside the moist room or curing tank for more than a few hours are much less suitable, because specimens that are cracked or that have slender cross sections sometimes carbonate very rapidly. Specimens exposed to simulated weathering tests, or wetting and drying cycles, or prolonged drying are not to be considered as representative of normally cured or of naturally weathered concretes. Natural weathering differs from part to part of a structure, as well as from climate to climate and subgrade to subgrade.

INTERPRETATION OF OBSERVATIONS

Normal Concrete:

For this discussion, "normal" constituents and fabrics are defined as those present in serviceable concrete of the class and age in the region. "Serviceable" is used instead of "undeteriorated" because it is possible to tell whether concrete in a structure is serving as it was intended to, but the criteria that distinguish inevitable chemical and physical changes from deterioration in concrete 20 or 50 years old have not been well established.

The most valuable information that can be obtained by petrographic examination of concrete comes from the examination of normal concrete; only by comparison with the range of constituents and fabrics in normal satisfactory concrete can that which differs from the normal be recognized and its differences specifically defined. Unless it can be demonstrated that the constituents, or the proportions of constituents, or the fabric, depart from those found in serviceable concrete of the age and class in the region, there is no logical basis for assuming any connection between constituents, or proportions, or fabric, and service behavior. Even when it can be shown that a concrete has a peculiar service record and some unique feature or features not shared by a dozen others of comparable class, age, and provenance, it remains to be seen whether the known unique feature and the peculiar service record are causally connected, or whether both are related to some third or *n*th factor that is the effective cause of the abnormal behavior.

Class of Concrete:

The restriction to concrete of one class is necessary because changes in cement content, water-cement ratio, and maximum size of aggregate large enough to change the class entail such large changes in properties that no close comparison will be significant. If, for example, the criteria for paving concrete are applied to mass concrete, all mass concrete appears very inferior, which it is not for the purpose it is intended to serve. Class of concrete is important in the definition as it implies relative homogeneity in mixture proportions, particularly in water-cement ratio, cement factor, and maximum size of aggregate. It is possible by microscopic methods to sort mass concretes that are fairly homogeneous in cement content and water-cement ratio in order of increasing age, or it is possible to sort mass concretes fairly homogeneous in age in order of increasing cement content. In terms of the ability to sort mass concrete microscopically, "fairly homogeneous" in cement content and water-cement ratio means a maximum difference in cement content between concretes of about 0.5 bags per cubic yard, and 0.1 by weight in water-cement ratio.

Age of Concrete:

Some restriction on the ages of concretes compared is necessary unless age is the variable being studied. Unless the age is known or unless one has younger and older concretes of otherwise comparable characteristics, so that the age of the unknown may be estimated in relation to the knowns, it may be impossible to judge the significance of observations. For example, in one case calcium sulfoaluminate was found in many voids as far as 5 in. from the outer surfaces of a concrete pavement of high flexural and compressive strength and of unknown age. In other field concrete from the region, calcium sulfoaluminate is commonly present in concrete over five years old made with Type I or Type III cement, but it is not abundant and is confined to voids near outer surfaces. If the concrete of unknown age is in fact five or seven years old, it differs conspicuously from others of comparable age and class in the region and the difference probably justifies some concern about its future; if it is 15 years old, it is peculiar, but the peculiarity is probably of less practical importance.

Provenance of Concrete:

Restriction to one region assists rational comparison from several points of view. The aggregates economically available in an area are determined by the regional geology and consequently show some homogeneity of composition resulting from similarity of origin and history. In a particular region, cements and aggregates economically available are used in making concrete which is exposed to the climate characteristic of the region—the prevailing temperature range and temperature frequency distribution and the characteristic amount and sequence of precipitation. The extent of a region of comparable concrete may vary from a few square miles to many thousands, depending on variation in: (1) *regional geology*—as it determines quantity and uniformity of aggregate supply; (2) *topography*—a region of low relief and generally uniform slope such as the Great Plains, or the Atlantic or Gulf Coastal Plain, has widespread, essentially comparable range and distribution of temperature and precipitation, but in a region of high relief and broken slopes, temperature varies considerably with altitude, and precipitation with orientation to prevailing winds, making important differences in exposure over short distances; and (3) *patterns of distribution of aggregates and cement from competitive sources*—in some areas, only one type of natural sand and gravel is available; no manufactured aggregate is produced, and synthetic aggregate sources are not common. Metropolitan marketing areas served by water transportation usually have available a selection of natural coarse and fine aggregates, manufactured coarse aggregates, and synthetic aggregates. All variations between the two extremes just mentioned can be found in availability of aggregates.

Cement plants per state range from none in New Hampshire and Vermont to 22 in Pennsylvania [*34*]. Ports and coastal areas may be served in periods of high construction activity by overseas and domestic cement sources.

An additional influence that may appear is a prevailing engineering opinion, in an organization placing concrete in a large area, on what is desirable in mixture proportions or methods of placing or consolidation. In building gravity dams the Corps of Engineers restricts the height of a single lift to 5 to 7½ ft [*35*] while the Hydroelectric Power Commission of Ontario has placed gravity dams up to 70 ft high in one continuous operation [*36*]. The intention in the first case is to minimize heat generation and in the second to eliminate horizontal construction joints. Such differences in emphasis entail differences in mixture proportions, plant, and construction practice that are bound to affect the fabric and character of the concrete. The existence of satisfactory structures built in many different ways underlines the need to define "normal" concrete in objective and restricted terms.

EXAMPLE

Although the most important kind of petrographic examination of concrete is the examination of normal concrete, usually the concrete that a petrographer is asked to examine has behaved in an unexpected way. Before and during the early stages of the examination, the information on the history and behavior should be considered and the following questions asked:

1. What process or processes could produce the described results?
2. What observable traces could the process or processes leave in the concrete?
3. Would such traces be unique and specific evidence of what is supposed to have happened?

Consider, for example, two 6 by 12-in. cylinders made at a field project during the winter; the 28-day strengths were 885 and 1025 psi, less than 25 per cent of the strengths obtained from earlier and later cylinders made, cured, and tested under presumably similar conditions; the resident engineer suspects an overdose of air-entraining agent.

Processes that Could Produce the Results:

Several distinct processes were considered that could have produced the observed results. Increasing the air content does reduce the compressive strength; the observed reduction is so drastic, however, as to require an increase in air content of at least 15 per cent of air to be a sufficient cause. Mistakes in batching, such as too little cement or too much water or damage due to early freezing, also could produce the observed results.

Traces that These Processes Could Leave:

Excessive air content can be recognized at low magnification and verified and quantified by comparison with concrete of known air content and by count. ASTM Recommended Practice for Microscopical Determination of Air-Void Content, Specific Surface, and Spacing Factor of the Air-Void System in Hardened Concrete (C 457 - 60 T) describes procedures for determining parameters of the air-void system.

Too little cement can be demonstrated by comparing thin or polished sections from cylinders of normal strength and the same mixture and age, with sections from these cylinders, and finding substantially less cement in the low-strength cylinders.

Too much water produces sedimentation even in air-entrained concrete, and may be demonstrated by comparing finely-ground surfaces of axial slices of cylinders of normal strength and those of reduced strength, and finding wider underside separations between paste and aggregate in the cylinders of reduced strength; the result may be inconclusive. More complete demonstration would result from comparing thin sections cut parallel with the placing direction from cylinders of normal strength, of the same mixture and age with sections cut in the same orientation from these cylinders, and finding probably a little less unhydrated cement with a large development of calcium hydroxide rims along the undersides of aggregate in the low strength cylinders.

Freezing before final set may leave imprints of ice crystals recognizable at low magnification. Freezing immediately after set may produce closely spaced fractures parallel to surfaces exposed to the low temperature [*37*].

Unique and Specific Evidence:

Lacking the necessary comparative material—cylinders of normal strength and similar age and history from the same mixture—it is very difficult to show petrographically that a large part of the cement was left out or that a large excess of water was put in. By making three sets of specimens from one mixture, one set with normal proportions, one with reduced cement, one with added water, curing all three in standard conditions, breaking part in compression, and sectioning companion specimens, a strong presumption might be established that one of the two working hypotheses was correct; it would not be conclusive proof. Axon's method [*38*] of determining original mixture composition, by micrometric determinations of aggregate content, paste content, and air content, supplemented by measurements of total evaporable water and evaporable water in aggregate, permits a calculation of cement content of the original concrete, if the age of the concrete can be approximated. Axon [*38*] and Mielenz [*4*] have used the method successfully.

Demonstration that the concrete froze before final set would depend on finding the imprint of the ice crystals. Excessive air content, on the other hand, can be qualitatively and quantitatively demonstrated without ambiguity.

NOTE—This is the surface as received, with most of the surface skin rubbed off in handling. The void walls can be broken with a fingernail The sides of the cylinder show the same condition with finer bubbles in some areas.

FIG. 1—Void size and distribution in the cement paste just inside the surface skin, bottom of low-strength cylinder (×6).

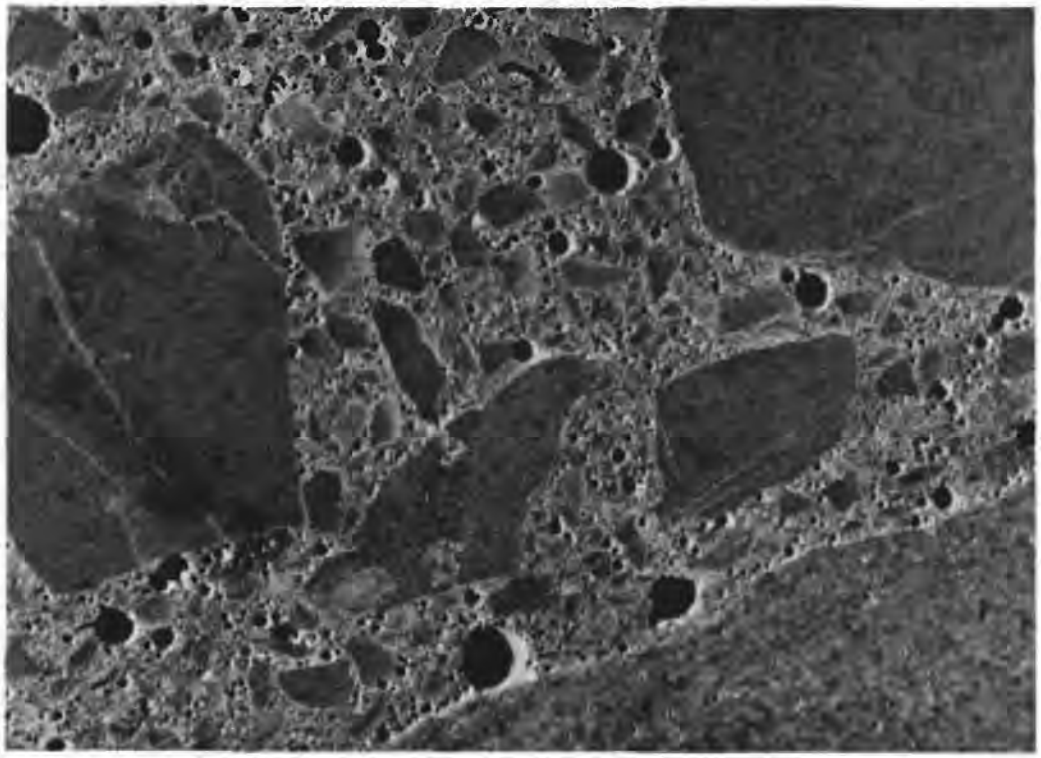
Let us hope that the resident engineer in our example is right, for the other possibilities that suggest themselves would be harder to establish. Examination of broken surfaces, outer surfaces, and sawed slices did show that the air content of the concrete was phenomenally high, around 25 per cent (Fig. 1) by comparison with concrete of known air content

(Fig. 2). The air content was so high and the thin walls of paste between the air voids so fragile that the low strengths were explained adequately by this evidence alone.

This example illustrates how one goes about asking relevant questions that can be answered by petrographic evidence. The general question in examination of hardened concrete with peculiar behavior is: "Does this concrete differ significantly from comparable normal concrete, with respect to one or more properties that may be shown to be causally connected with its behavior?" The generalized form of the null hypothesis is that the concrete falls within the normal range in respect to a certain property or properties.

Of the four mechanisms considered, three should produce recognizable traces in amounts that can at least be approximated. The freezing-before-final-set hypothesis was poorly defined in effects and their magnitude. The ability to be quantitative and the ability to obtain confirmation by nonmicroscopic means differed in the three hypotheses. Air content is specified in a numerical range, in this case, 4 to 7 per cent measured at the mixer.

Brown and Pierson [*39*] have shown and others confirmed that measurements of the air content of freshly mixed concrete and micrometric determinations of the air content of the same concrete allowed to set undisturbed agree very closely. If concrete with 7 per cent air



NOTE—The air content of the plastic concrete determined by pressure meter was 5.3 per cent; the air content of this hardened beam by micrometric count was 5.7 per cent. This surface is smoother than the surfaces in the other photograph, but this concrete obviously has less than a third of the air content of the other.

FIG. 2—This diamond-sawed and ground slice of air-entrained concrete provides a comparison with Fig. 1 (×6).

were produced and if a generous allowance for field and laboratory error is made, it would still be most surprising to find more than 10 per cent air in a supposedly normal cylinder from this project. The low strengths were in the range of strengths of moist-cured foamed neat cement [*40*]. With this background we conclude that unless the low strength cylinders have a total air content above 20 per cent, the excessive air hypothesis could not be accepted as an adequate explanation of the strengths. The hypothesis can be quantitatively expressed, and the quantities can be measured in more than one independent way; the magnitudes involved can be distinguished by anyone who can see through a stereoscopic microscope and ask, "What relation does the air content of A bear to the air content of C?"

The quantitative aspects of both forms of the batching-mistakes hypothesis are not so well explored, and data are less easily verified by independent means. The volume of cement in the original mixture and the volume of calcium hydroxide at a given stage in hydration can be calculated making certain assumptions, which in this example could not be confirmed. However, given comparable cylinders of normal strength, the omitted-cement hypothesis could be checked by comparing sections from the normal concrete with sections from the low-strength concrete to see whether there was a difference between the amounts of unhydrated cement per unit area. If the difference were to provide a satisfactory explanation, it would be fairly large and should be perceptible to an observer able to recognize unhydrated cement in thin or polished sections. Some petrographers might choose to count a group of 300 to 500 points on a section of each kind in order to obtain a result that can be expressed as a number, with the understanding that the sample was not adequate; others would prefer to look at several areas and express the results as "more than . . ." "less than . . . ," or "no difference recognized." The procedure in checking the extra-water hypothesis is similar but would concern the development of calcium hydroxide rims along undersides of fine aggregate.

The simple, orderly example just described involves a problem that can be solved without techniques more elaborate than those normally used in several concrete laboratories in this country in 1945. The simplicity and order arise because the concrete examined was two test specimens, and both the history available and the total age of the specimens made it clear—and examination of the concrete adequately confirmed—that no chemical attack on cement paste had taken place and that reactions between the particular aggregates and cement could not have developed, in the time available, to a degree that could render them significant effects on the strength.

RECONSTRUCTION OF HISTORY OF FIELD CONCRETE

To pass from consideration of such petrographic examinations as that described in the previous example to the petrographic examination of concrete that has aged and perhaps deteriorated in service, introduces two important new unknowns—time and the precise environment of the structure. The effects of the passage of relatively short periods of time on the constituents present in several cement pastes of known water-cement ratio stored under laboratory conditions have been investigated [*41,46*], but anomalies remain [*43*] in the results, although both the composition of the pastes and the nature of the environments were known and controlled far more thoroughly than the composition and environment of any field concrete.

Composition:

If the changes in composition through time of cement paste in laboratory conditions were known for a representative number of cement compositions and water-cement ratios, effects of both cement-aggregate interactions and of environmental influences would be easier to recognize and could be more usefully interpreted.

Environment:

Why do exposed vertical walls of chert-gravel concrete in the vicinity of St. Louis, Mo., generally have fewer popouts than apparently similar walls in the vicinity of Memphis, Tenn.? The winters are colder in St. Louis, but the mean annual rainfall is lower; and in Memphis a larger proportion of the higher mean annual rainfall occurs in winter. The difference may be that the chert gravel in the Memphis walls is more likely to be critically saturated when it freezes. The Weather Bureau's climatological data for the location are a valuable source of information that can assist in many petrographic examinations of hardened concrete.

The discovery in Mississippi of several highway pavements and associated structures affected by sulfate attack and by combined sulfate and acid attack [*47*] emphasizes the need to make use of available information on the composition of foundations and subgrades.

Thus, the examination of samples of field concrete after extended service involves an increase in complexity, a decrease in available information, and a decrease in the confidence that may be placed in the answer, as compared to examinations of laboratory test specimens of hardened concrete.

It does not belittle the petrographer to admit that he cannot make bricks without straw; neither can the chemist, the physicist, or the engineer; sometimes the petrographer can recover evidence not accessible by other approaches. Several reasons make reconstructions of the history of deteriorated field concrete difficult; in any particular instance it may be important and impossible in the present state of our knowledge to decide what weight belongs to each.

Deteriorated field concrete that is referred to a laboratory or to a petrographer is almost never concrete that has performed abnormally for one single obvious cause; such simple cases can be and are explained on the spot to the satisfaction of those concerned. The field concrete that is examined by a petrographer is concrete that has worried some responsible person enough to make the effort and expense of sampling and testing appear justified. There is thus a built-in bias in the sampling process. Normally the concrete that a petrographer sees as part of his assigned duties is controversial concrete sent in by organizations with alert conscientious concrete technologists. In practice, this generally means that he sees only the poor concrete produced with better than average control. The worst concrete is rarely sampled and sent to a petrographer; good concrete is rarely controversial.

Furthermore, the older the concrete the less information is likely to be available about materials, proportions, conditions of placing, and the characteristics that undeteriorated comparable concrete would have. Although one can deduce from the concrete that water-cement ratio was high or low, and usually that cement factor was high or low or medium, and the general quality of the workmanship, one cannot reconstruct the alkali content of the cement. Aggregate sources, particularly of natural sand and gravel, can be located from their composition—the constituents present and their size distribution are diagnostic of the region and sometimes of the particular source.

Finally, deteriorated field concrete usually shows superimposed traces of several processes, with at least one in an advanced stage. The most advanced process may conceal the evidence of others that were more important in effect. Frequently the most conspicuous process is carbonation of outer surfaces and along the borders of old cracks.

Laboratory test exposures are simplified compared to natural exposures by the exclusion of some factors and the regulation of those retained and often are "accelerated" by altering some factor so as to remove it from the range possible in nature. Consequently, a laboratory procedure often results in symptoms different from symptoms encountered in a field example of the process the test is intended to simulate.

Samples of field concrete, when examined using light microscopy, frequently are found to contain secondary calcium carbonate near their outer surfaces, along old cracks, and sometimes in the interior. Such calcium carbonate, when examined by optical methods, is generally found to be calcite, rarely aragonite, and almost never vaterite, the form-birefringent spherulitic calcite with interstitial water. Vaterite, however, was found by optical methods to be common on mortar bars that had been tested according to ASTM Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227) and had been found on concrete specimens tested for resistance to freezing and thawing according to ASTM Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290 – 63 T). The use of X-ray diffraction to examine cement-paste concentrates from field concrete has revealed that vaterite, not recognized by optical methods, is frequently a major constituent of the secondary calcium carbonate [*48*], especially on samples from seawater exposures or from other wet environments. Vaterite is known to persist for several months in laboratory specimens stored in room conditions. The sequence from poorly crystallized vaterite, calcite, and aragonite to well-crystallized calcite in the carbonation of pastes and mortars has been clarified by Cole and Kroone [*49*], and vaterite is now known as a natural mineral [*50*].

Accelerated freezing and thawing in water according to ASTM Method C 290 produces a characteristic loss of surface skin and loss of mortar, which is not like the condition of specimens exposed on the mean-tide rack at Eastport, Me. [*51*]. Some field concrete, deteriorated by natural freezing and thawing, develops sets of subparallel cracks normal to the placing direction of the concrete or deteriorated regions parallel to the nearest free surface. These phenomena are not reproduced in accelerated freezing and thawing in water.

Field concrete that has deteriorated primarily because of the alkali-silica reaction usually has much more advanced and conspicuous internal symptoms of this reaction than are found in mortar bars of expansive combinations examined after test according to ASTM Method C 227. On the other hand, some field concrete regarded as undeteriorated has shown a range of evidence of alkali-silica reaction.

Alkali-carbonate and alkali-silica reactions exist together in varying degrees of development in some concretes [*18*], and inconspicuous degrees of reaction may be the only recognizable peculiarities in cases of unsatisfactory service with possibly expensive consequences [*52*]. The several alkali-carbonate reactions [*53*] include dedolomitization and rim-forming reactions [*17,53,54*]. The effects of rim-forming reactions are as yet inadequately known, although they appear to reduce strength [*53*].

CLOSURE

It was true in 1955 and still is that we do not yet know enough about the hydrous calcium silicates that hold concrete together, the other compounds associated with them, and the varying successions of compounds in laboratory and field hydration of concrete. For instance: hexacalcium aluminate trisulfate hydrate [*55*]—more usually known as "high sulfate sulfoaluminate" or ettringite—is a normal early hydration product of most cements. It is not stable in the presence of tricalcium aluminate when hydration can continue, and frequently disappears from cement pastes after longer curing. If it is found in a 20-year old concrete, does it indicate some abnormality? Calcium aluminate monocarbonate (or monocarboaluminate) [*15*,*55*] is found in laboratory cement pastes and in field concrete [*18*,*48*]. In the laboratory it can be produced by accidental carbonation or by the addition of finely ground calcite to the freshly mixed paste [*55*]. Hadley [*53*] recognized it at the interface of dolomitic limestone and cement paste in dedolomitization. What is its effect in field concretes with and without carbonate aggregates [*56*]?

The measure of progress and the results of the use of newer techniques including X-ray diffraction, differential thermal analysis, electron microscopy and electron diffraction are that the questions listed above, and others, are obvious to me in 1965, although I could not have formulated them in 1955. "Things which are seen"—concrete and mortar—"were not made of things which do appear" [*57*] to the eye and to the light microscope. The use of the newer techniques mentioned and of the electron probe and infrared spectroscopy, in conjunction with the observing eye and the light microscope, still offers the chance of sorting out the qualitative and quantitative differences in hydration products and in submicroscopic fabric that are related to serviceable and deteriorated concrete.

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STP169A-EB/Apr. 1966

Hardened Concrete

STRENGTH

By C. E. Kesler,[1] Personal Member, ASTM

Tests to determine strength are undoubtedly the most common type made to evaluate the properties of hardened concrete. There are three reasons for this: (*a*) the strength of concrete, in compression, tension, shear, or a combination of these, has in most cases a direct influence on the load-carrying capacity of both plain and reinforced structures; (*b*) of all the properties of hardened concrete, those concerning strength can usually be determined most easily; and (*c*) by means of correlations with other more complicated tests, the results of strength tests can be used as a qualitative indication of other important properties of hardened concrete.

The list of references at the end of the paper is not intended to be complete. The references chosen are those considered to be most useful and readily available to the reader and preference is given to the latest papers. Two excellent references for strength of concrete under static loads are by Price [*1*][2] and Neville [*2*].

Compressive Strength

Significance of Compressive Strength:

The compressive strength of concrete is one of its most important and useful properties and one of the most easily determined. In most structural applications, concrete is employed primarily to resist compressive stresses. In those cases where strength in tension or in shear is of primary importance, the compressive strength is frequently used as a measure of these properties. Similarly, the compressive strength is used as a measure of the over-all quality of the concrete and thus as an indication of other properties relating to deformations or durability. And finally, the concrete-making properties of the various ingredients of the mix are usually measured in terms of the compressive strength.

Specimens:

Specimens to determine the compressive strength of concrete are generally obtained from four different sources: (*a*) cylinders made in the laboratory, (*b*) cylinders made in the field, (*c*) cores of hardened concrete cut from structures, and (*d*) portions of beams broken in flexure. Each type of specimen has a specific purpose or purposes.

Cylinders made in the laboratory constitute a large portion of the compression specimens. Their purpose is threefold: (1) in research, to determine the effect of variations in materials or conditions of manufacture, storage, or testing on the strength and other properties of concrete; (2) as control tests in conjunction with (*a*) tests on plain or reinforced concrete members or struc-

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

tures or (*b*) tests to determine other properties of hardened concrete; and (3) to evaluate mix designs for laboratory or field use. The ASTM Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192) describes in detail methods for preparation and examination of the constituent material; proportioning and mixing of concrete; determining the consistency of the mix; and molding, curing, and capping of the specimens. The usual compression specimen is a cylinder having a height twice its diameter.

The procedure for making standard cylinders in the field is described in the ASTM Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31). The purpose of cylinders made in the field may be to check the adequacy of the laboratory mix design, to determine when a structure may be put in service, or to measure and control the quality of the concrete. The curing used will depend on the purpose of the test but should reflect field conditions.

Cores are often drilled from hardened concrete when the results of the standard cylinder tests are questionable or when investigations are made of old structures. The ASTM Methods of Obtaining and Testing Drilled Cores and Sawed Beams of Concrete (C 42) covers the procedure for securing and testing the cylindrical cores which are most commonly used for determining compressive strength. When possible, the core should be cut in such a manner that when the test load is applied it will act in the same direction as the service load. The ratio of height to diameter of cores may vary, and only seldom will it be possible to obtain a height to diameter ratio of two. ASTM Methods C 42 gives the following correction factors for converting the strength of a test core to that of a standard cylinder with a height to diameter ratio of two.

Ratio of Length of Cylinder to Diameter	Strength Correction Factor
1.75	0.98
1.50	0.97
1.25	0.94
1.00	0.91

These correction factors apply to lightweight as well as ordinary concrete. They are not necessarily applicable to autoclaved concrete or to concrete dry at the time of loading. These correction factors are generally applicable for nominal concrete strength from 2000 to 6000 psi (1400 to 4220 kg/cm^2). The magnitude of the correction required is dependent on the age, strength, mix, and moisture content of the core at the time of test [*3*].

Most recent of the various methods for determining the compressive strength of concrete is that employing portions of beams broken in flexure. It is particularly useful in cases where beam specimens are made to determine the modulus of rupture, as in highway construction, since the broken portions of these beams may be used to determine the compressive strength. The ASTM Test for Compressure Strength of Concrete Using Portions of Beams Broken in Flexure (C 116) describes the procedure and apparatus necessary to determine compressive strength by this method. The projection of the beam beyond the loading plates in this test may affect the strength, yielding a lower value if the projection is less than 2 in. (5 cm); if the projection is greater than 2 in. (5 cm), there will probably be no significant effect. It would be desirable to have a correction factor. Method C 116 states that the results of these tests are not expected to correlate with those of cylinder tests. Results of investigations in general do not agree on any one correlation factor between these tests and standard cylinder strengths [*4*]; however, it should be possible, for a specific set of circumstances, to obtain and use a specific correction factor.

Making Specimens:

The compressive strength is affected by many variables encountered in the making of specimens. These include size of aggregate, size and shape of specimen, compaction of concrete, type of mold, capping procedure and material, curing, temperature, and moisture content at the time of test.

It is generally accepted that the diameter of the specimen should be at least three times the nominal size of the coarse aggregate. A 6 by 12-in. (15.24 by 30.48 cm) cylinder is the standard for aggregate smaller than 2 in. (5.08 cm). If the aggregate is too large for the size of mold available, the oversize aggregate may be removed by wet screening [*5,6*]. If a mold having a diameter less than three times the maximum size of the aggregate is used, the indicated compressive strength will be lowered. However, a larger mold may be used; in some cases molds as large as 36 in. (91.44 cm) in diameter have been used for concretes containing very large aggregates, such as those used in dam construction. Attention must be called to the fact that the size of the cylinder itself affects the observed compressive strength; for example, the strength of a cylinder 36 in. (91.44 cm) in diameter by 72 in. (182.88 cm) high may be only about 82 per cent of that of a standard 6 by 12-in. (15.24 by 30.48-cm) cylinder. A reduction in the size of the specimen below that of the standard 6 by 12 in. (15.24 by 30.48-cm) cylinder will yield a

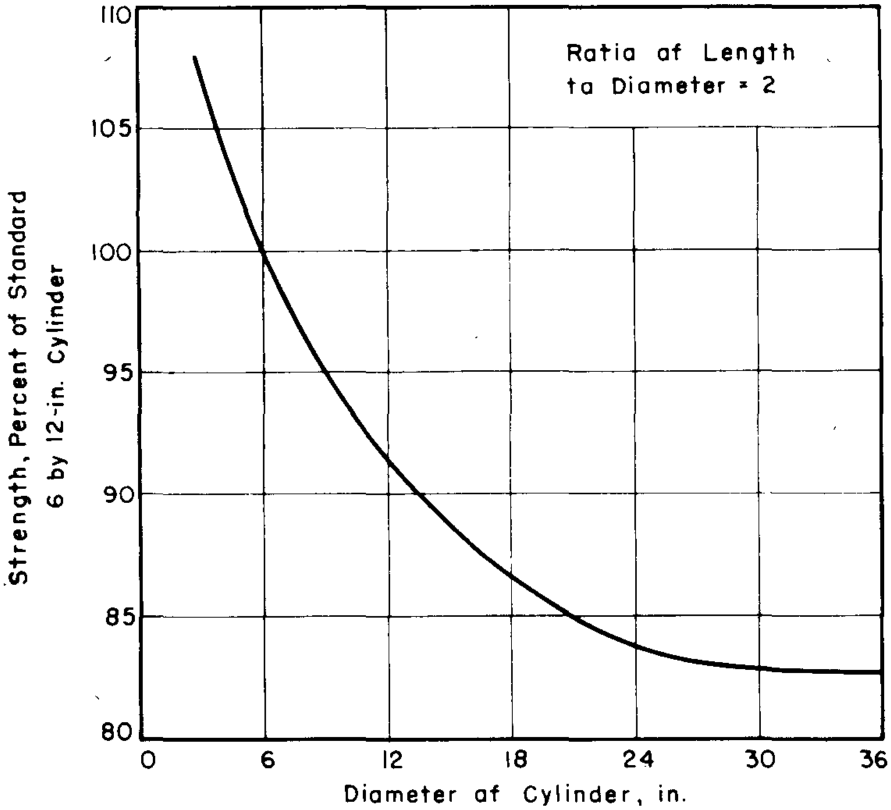

FIG. 1—Effect of diameter of cylinder on compression strength.

somewhat greater indicated compressive strength, Fig. 1 [*1*].

Cylinder molds should be of nonabsorbent material and are generally of steel; however, cardboard molds are quite often used in the field. The ASTM Specification for Single-Use Molds for Forming 6 by 12-in. (15.24 by 30.48-cm) Concrete Compression Test Cylinders (C 470) defines adequate paper molds as well as lightweight sheet steel molds. Although the cardboard is heavily paraffined, in most cases it absorbs part of the water in the concrete mixture. The use of cardboard molds may lower the observed compressive strength on the average about 3 per cent, and reductions as great as 9 per cent have been noted [*1,7*]. The exact reason for this is not known, although it may be due partly to expansion of the mold during the setting period of the concrete as the result of absorption of water from the mix by the cardboard.

Other things being equal, a cylinder of poorly compacted concrete will have a lower strength than one that is properly compacted. Thus, it is necessary for the standard methods for making specimens, Methods C 31 and 192, to specify procedures for compacting, rodding, or vibrating the concrete in the mold. If the specifications under which the work is being done do not state the method of consolidation then the choice is determined by the slump. Concrete, with a slump greater than 3 in. (7.62 cm), should be rodded. If the slump is between 1 and 3 in. (2.54 and 7.62 cm), the concrete may be either vibrated or rodded. When the slump is less than 1 in. (2.54 cm), the specimens must be consolidated by vibration. When the concrete is to be rodded it should be placed in the cylinder in three layers and rodded 25 strokes per layer if the cylinder is 3 to 6 in. (7.62 to 15.24 cm) in diameter, 50 if 8 in. (20.32 cm), and 75 if 10 in. (25.40 cm). When vibration is used, the mold is filled and vibrated in two layers. Care must be taken to vibrate only long enough to obtain proper consolidation. Over vibration tends to cause segregation. These methods are specified in order to permit reproducibility of results by different technicians.

Capping is one of the most important steps in the preparation of test specimens. Whether cylinders of portions of beams are to be tested the loaded surfaces must be plane within 0.002 in. (0.005 cm). The capping material may be neat portland-cement paste, high-strength gypsum plaster, or sulfur compounds [*8,9*]; in some cases the ends of the specimens may be ground to a smooth surface. The cap should be as thin as possible and must be at least as strong as the concrete. After capping, the ends of the specimen should be parallel, perpendicular to the axis of the specimen, and plane. If the ends are more than 3 deg out of parallel, the observed strength may be adversely affected, and only a slight amount of convexity or concavity of the ends is sufficient to cause a decrease in strength of the test specimen [*10,11*].

Concrete can gain in strength only as long as moisture is available and used for hydration. The standard curing conditions require that the specimen be held at a temperature of 73.4 ± 3 F (23 ± 1.7 C) and in the "moist condition" until the time of the test. "Moist condition" is defined as that in which free water is maintained on the surfaces of the specimen. Any variation from this procedure may produce a specimen having a different strength from that which would be produced under standard conditions.

Cylinders to be used for quality control should be cured according to the standard conditions; however, cylinders made in the field and tested to measure the strength of the concrete in the structure should be cured in the same manner as the structure. Concrete cores are generally taken from partially dry concrete, but the moisture content may vary considerably. To obtain a degree of uniformity in the test specimen at the time of test, Methods C 42 states that cores be soaked in water for at least 40 hr immediately prior to being tested and that they be tested wet. Under certain conditions the agency for which cores tests are being made may direct that specimens be tested in a moisture condition other than that achieved by soaking. The standard test for cylinders, ASTM Test for Compressive Strength of Molded Concrete Cylinders (C 39) also requires that they be tested wet. If other conditions are equal, the compressive strength of concrete cylinders or cores tested dry will be greater than that of comparable specimens tested wet [*12*,*13*].

Curing temperature affects the rate of hydration of concrete; therefore, the temperature of curing under standard conditions is specified as mentioned previously. Generally speaking, low curing temperatures tend to give low strengths whereas high curing temperatures give higher strengths, provided the specimens are cast at the curing temperature. If the concrete is cast at a temperature lower than the curing temperature, the resulting observed strength will be higher than that obtained if both temperatures are the same, and *vice versa* [*1*,*14*].

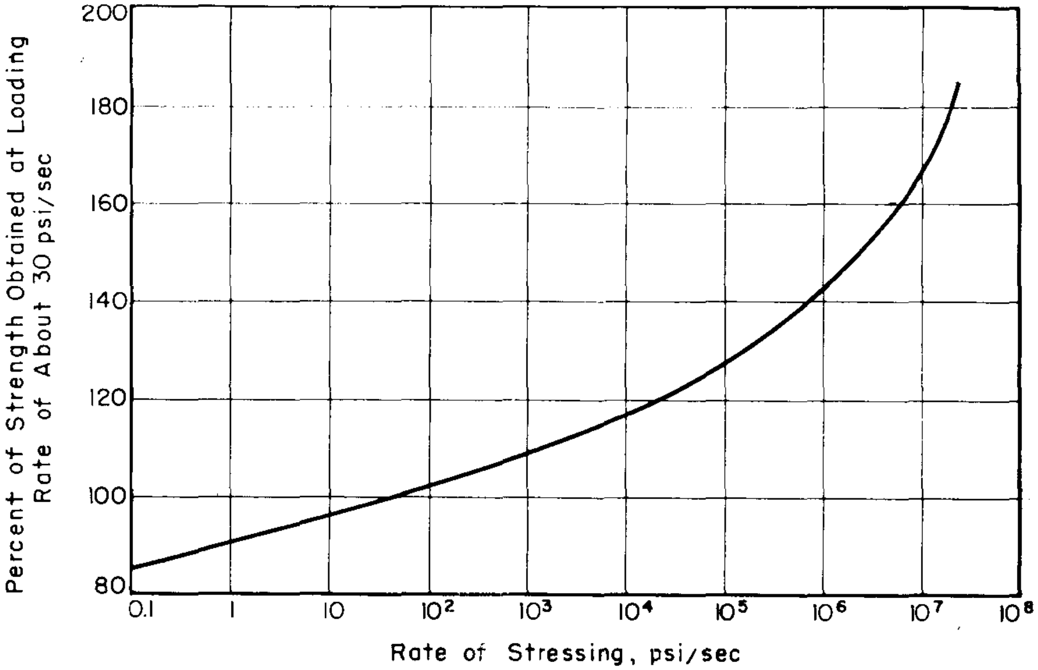

FIG. 2—Effect of rate of stressing on the compressive strength of concrete.

Test Procedure:

Once the specimen is made, the method by which it is tested may further affect the strength obtained. Some of the more important influences are the rate of loading, the eccentricity of loading, and the temperature at the time of test.

The apparent strength of concrete increases as the rate of loading is increased, Fig. 2 [*15*,*16*]. For this reason the ASTM Method C 39, which applies also to tests of cores, specifies that the rate of loading for screw-powered machines shall be 0.05 in. (0.13 cm/min) and for hydraulic machines 20 to 50 psi/sec (1.41 to 3.52 kg/cm^2/sec). However, the first half of the load may be applied at a faster rate.

To insure that a concentric and uniformly distributed load is applied to the specimen, a spherically seated bearing block is required on one end, and the specimen should be carefully centered on this bearing block. Any eccentricity will tend to decrease the strength of the test specimen, the amount of decrease being greater for low-strength than for high-strength concretes [*10*].

The temperature at the time of test influences the compressive strength. As the temperature is increased, the strength obtained in the test is decreased [*2*]. For the range of temperature normally encountered indoors, however, this effect is probably negligible.

Significance of Results:

The compressive strength of concrete as determined from a standard cylinder which may be cured in the same manner as the concrete in the structure will give an indication of the quality of the concrete in the structure but not necessarily the strength. The strength of concrete in a structure may be different from that of the same concrete in a cylinder because of differences in size, shape, position of casting, degree of compaction, conditions of restraint, curing conditions, and variations in moisture content. Lack of knowledge regarding the relationship between the strengths of concrete in a cylinder and in a structure requires the use of a larger factor of safety than would otherwise be necessary.

The compressive strength of concrete under long-sustained load is less than that determined by the standard short-time test method [*1,17*]. Tests indicate also that a sustained stress less than about 70 per cent of the short-time strength will have little effect on the compressive strength developed in a subsequent short-time static test [*18*].

Compressive strength may be used also as a qualitative measure of other properties of hardened concrete. No exact quantitative relationships between compressive strength and flexural strength, tensile strength, modulus of elasticity, wear resistance, fire resistance, or permeability have been established nor are they likely to be. However, approximate or statistical relationships [*19*] in some cases have been established, and these give much useful information to the engineer. It should be emphasized that the compressive strength gives only an approximation to the values of these properties and that other tests specifically designed to determine these properties should be used if more precise results are required. For instance, the indicated compressive strength increases as the specimen dries, whereas a modulus of rupture specimen decreases in strength.

Compressive tests aid in the selection of ingredients that may be used in making concrete. Compressive strength is a measure of the indirect effect of admixtures which may be beneficial for one purpose, such as waterproofing, but may be detrimental to another property. In evaluating the efficiency of concrete mixers, the compressive strength test is frequently the principal criterion used and sometimes the only one.

TENSILE AND FLEXURAL STRENGTH

Significance of Tensile and Flexural Strength:

Flexural tension is most commonly developed in beams and slabs as the result of loads, temperature changes, shrinkage, and in some cases moisture changes. The case of simple uniaxial tension is rarely encountered in structures or members and can be obtained in laboratory tests only with care. However, significant principal tension stresses may be associated with multiaxial states of stress in walls, shells, or deep beams.

Concrete is weak in tension, the tensile strength usually being between one eighth and one twelfth the compressive strength. Cracks occurring in concrete are caused by tensile failures, and this alone makes the tensile strength of concrete quite important. Failures in nonreinforced concrete beams are necessarily tensile failures.

Specimens:

No standard test has been developed for axial tension tests of concrete. However, for research purposes, concrete has been tested for tension in many ways: in the form of large briquets [*20,21*], with clamps on the ends of cylindrical specimens [*22*] or on specimens with enlarged ends [*23,24*], and with the load applied to the concrete through embedded bars [*25*]. Doubt about the uniformity of stress distribution in many of these tests causes the results to be regarded with suspicion. Tension tests made by the methods indicated are difficult to perform, and the individual results may vary considerably. The beam test for flexural tension and split cylinder test are the simplest procedures for obtaining an indication of the tensile strength.

Flexural tension tests may be made in several ways, the most common being the ASTM Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78) and the ASTM Test for Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading) (C 293). The second method is for small specimens and is not an alternate to the first which is normally for beams 6 by 6 in. (15.24 by 15.24 cm) or larger in cross section. The results of the flexural tests are expressed by the formula:

$$R = \frac{Mc}{I}$$

where in consistent units:
R = the modulus of rupture,
M = the maximum bending moment,
c = one half the depth of the beam, and
I = the moment of inertia of the cross section.

This expression is based on the assumption that the stress varies linearly across the cross section. Such is not the case, however, and the modulus of rupture computed from the formula is always greater than the actual stress.

Another procedure for obtaining an indication of tensile strength is given in ASTM Test for Splitting Tensile Strength of Molded Concrete Cylinders (C 496 – 64 T). In this test a standard cylinder is loaded in compression on its side. Fracture occurs along the plane which includes both lines through which the load is applied. While high compressive stresses occur at the lines where load is applied, the plane on which fracture occurs is subjected largely to a uniform tensile stress. The splitting tensile strength may be calculated as follows:

$$T = \frac{2P}{\pi l d}$$

where in consistent units:
T = the splitting tensile strength,
P = the maximum applied load,
l = the length, and
d = the diameter.

The theory on which the above expression is based assumed that the concrete is elastic and that a state of plane stress exists. Neither assumption is true and the calculated splitting tension test is slightly higher than the true axial tensile strength [*26*].

Making Specimens:

ASTM Methods C 192 and C 31 prescribe the procedures for making flexural test specimens and also cylinders for the splitting tensile strength test in the laboratory and in the field. The specimens should have a cross section with a minimum dimension at least three times the maximum nominal size of the coarse aggregate. The span of the specimen should be at least three times the maximum dimension of the cross section. A commonly used specimen is 6 by 6 in. (15.24 by 15.24 cm) in cross section, 21 in. (53.34 cm) long, and is tested under third-point loading on a span of 18 in. (45.72 cm). As the depth of the beam is increased, there will be a decrease in the modulus of rupture [*27–29*].

The method by which the concrete is compacted may affect its strength [*29*]; consequently, the ASTM standard methods specify the manner in which the concrete should be placed in the molds. Generally molds 8 in. (20.32 cm) or less are filled in two equal layers and those over 8 in. (20.32 cm) in three. The criteria for determining whether cylinders should be rodded or vibrated also apply to beams.

Curing affects the tensile strength in much the same manner as it affects the compressive strength [*22*]. A beam which has been allowed to dry during the curing or storage period will have tensile stresses in the outer fibers as a result of differential volume changes due to nonuniform drying (*30*). For this reason, beams tested "dry" usually yield lower flexural strength than those tested in the saturated condition. Consequently, in tests to determine or control the quality of concrete, uniformity of results will be assured only if the beams are cured in the standard manner and tested wet. Beams tested to determine when a structure should be put into service should be cured as nearly as practicable in the same manner as the structure but tested wet.

Test Procedure:

Flexural strength measurements are extremely sensitive to all aspects of specimen preparation and testing procedure [*31*].

The arrangement of the loading device and beam supports in the flexure test must be such that only transverse loads are applied [*32*]. Beams may be tested under either center-point or third-point loading. Third-point loading invariably gives lower strengths than center-point loading. Tests indicate the following order of decreasing magnitude of the strength obtained: (1) center loading, with moment computed at center; (2) center loading, with moment computed at point of fracture; and (3) third-point loading [*28,33*]. Third-point loading probably gives lower strengths because the maximum moment is distributed over a greater length of the beam; since the concrete is not homogeneous, this loading method seeks the weakest section.

The rate of load application, unless standardized, may cause considerable variation in the results of flexure tests, the variation being as much as 15 per cent for the range of rates that may be obtained in the average laboratory. The higher rates of loading give higher strengths [*16,28*].

The moisture content of the specimen at the time of test affects the strength determined, as discussed in the preceding section. Flexural specimens are so sensitive to moisture content that ASTM standard methods for making them require that they be put in saturated lime solutions at least 20 hr immediately prior to testing, regardless of whether the specimens are to check quality or determine when a structure may be put into service.

The temperature of a beam specimen at the time of test will affect the results. As the temperature increases, the strength decreases [*2*].

ASTM Method C 78 specifies that the specimen shall be turned on its side before being tested. If the molded sides are plane and parallel, the specimen then does not need to be capped; however, if the molds are not satisfactory, the specimen must be capped.

ASTM Method C 78 is also prescribed for tests of beams sawn from hardened concrete. When such beams are used primarily as a control of concrete quality, they should be turned on their sides before testing and will usually require capping because of the irregularity of the sawn surfaces. In special cases where sawn beams are tested to provide specific information regarding the strength or behavior of members from which they are obtained, it may be desirable to test them in the as-cast or inverted position rather than on their side as required by Method C 78. However, there is no assurance that the results of such tests can be correlated with data obtained from beams tested in accordance with Method C 78.

The test for splitting tensile strength described in Method C 496 is simple to make. The effectiveness with which the material in the bearing strips is able to conform to the irregularities of the surface of the specimen and distribute the load affects the results. For uniformity Method C 496 specifies that $\frac{1}{8}$-in.-(0.32-cm-)thick plywood shall be used for bearing strips. Care must be taken to apply the load through a diametrical plane. As with other strength tests of concrete, the rate of loading affects the results. The load should be applied such that the stress increases between 100 and 200 psi/min (7.03 and 14.1 kg/cm^2).

Significance of Results:

Flexural test results appear to be most useful in pavement construction. In such cases, the flexure test rather than the standard compression test is frequently used to determine the quality of the mix. Many agencies do not even make compression tests in connection with their pavement projects. The strength obtained from flexure tests is not necessarily a precise measure of the strength of the concrete in the structure, even when the curing conditions are duplicated. Such tests are quite useful, however, as an indication of when the concrete has gained sufficient strength that load may be applied or the forms removed.

The splitting tension test has been used to evaluate bond splitting resistance of concrete. The American Concrete Institute Standard 318-63, Section 505, Building Code Requirements for Reinforced Concrete specifies this test for evaluation of lightweight concrete. For this purpose the specimen is not moist cured and tested wet but is moist cured for 7 days followed by 21 days drying at 73 ± 3 F (23.0 ± 1.7 C) and 50 ± 5 per cent relative humidity. The specimen is tested in this air-dry condition.

With increased emphasis on control of cracking in reinforced concrete an appreciation of the tensile strength has become more important. However, the three general methods of estimating tensile strength give slightly different results. The splitting tensile strength is the easiest to perform and gives the more uniform results. The tensile strength from the splitting tests is about $1\frac{1}{2}$ greater than obtained in a direct tension test and about $\frac{2}{3}$ of the modulus of rupture [*26*].

SHEARING AND TORSIONAL STRENGTH

Significance of Shearing Strength:

The importance of shearing strength as a property of concrete is evident from the fact that the standard test cylinder, tested in axial compression, nearly always fails by shearing along an inclined plane. This type of failure, however, occurs as a result of a complex combination of normal and shearing stresses on the plane and is discussed further in the section on Combined Stresses. The case of pure shear acting on a plane is seldom if ever encountered in actual structures. Nevertheless, it is discussed briefly here in order to provide a better understanding of the problems encountered in attempts to determine shearing strength.

Pure Shear:

The resistance of concrete to pure shearing stress has never been directly determined. Whenever a state of pure shearing stress is produced in a specimen, it follows from the laws of mechanics that principal tensile stresses, equal in magnitude to the shearing stresses, must also exist on another plane. Since the strength of concrete in tension is less than its strength in shear, failure inevitably occurs as a result of tensile stresses before the strength in shear is reached. Thus, a pure shear test is consequently of no value for determining shearing strength.

Tests intended to provide data on strength in pure shear have been made on beams or slabs of very short spans with the load applied very close to the supports [*34*]. In some of these tests, the shearing strength has been found to be only slightly greater than the tensile strength, probably because tensile stresses were present on the plane of failure as the result of bending, or because of local crushing due to high bearing stresses. In other tests of this type, the shearing strength was found to be quite large, as much as 50 to 90 per cent or more of the compressive strength. In these cases, it seems likely that compressive stresses were present on the plane of failure.

A reliable indication of the strength of concrete in pure shear can be obtained only from tests under combined stresses, as discussed in the section on Combined Stresses.

Torsion:

The application of torsion alone to a concrete specimen produces pure shearing stresses on certain planes. However, as has been discussed previously, failure under these conditions will occur in tension rather than in shear. The strength of concrete subjected to torsion is related, therefore, to its tensile strength rather than to its shearing strength. If the specimen is a hollow cylinder, for which the tensile stresses are distributed nearly uniformly over the cross section, the tensile strength determined from a torsion test will usually agree quite closely with that obtained from direct tension tests. However, if the test specimen is a solid cylinder, the tensile stress at failure, computed by means of the conventional torsion formula assuming a linear stress distribution, will be greater than the tensile strength obtained from axial tension tests since the actual stress distribution is nonlinear.

COMBINED STRESSES

Significance of Combined Stresses:

Concrete in structures is almost never subjected to a single type of stress. Just as nearly all structural members are acted upon by various combinations of moment, shear, and axial load, the concrete in them is usually subjected to some combination of compressive, tensile, and shearing stresses.

Tests:

There is no standard test for determining the strength and mode of failure of concrete subjected to combined stresses. For purposes of research, relatively extensive tests have been made on cylindrical concrete specimens under triaxial compression [*35–37*]. Tests have also been made with combinations of axial tension and lateral compression [*25*], and torsion and axial compression [*38*].

Results of Tests:

There is no universally accepted criterion of failure for concrete; hence, there is no single correct way of discussing the behavior of concrete under combined stresses. While perhaps not the most accurate, a convenient method of interpreting the results of tests under combined stresses is by the Mohr rupture diagrams, Fig. 3.[3] The results of any given test under combined stresses can be represented on this diagram by the conventional "Mohr's circle," corresponding to the state of stress in the concrete at failure. For example, Circle *a* represents the result of an unconfined compression test, such as the standard cylinder test; Circle *b* is for an axial tension test; Circle *c* is for the case of pure torsion; and Circle *d* is representative of those obtained in tests under triaxial compression. The results of tests under combined torsion and axial compression would be represented by circles intermediate between Circles *c* and *a*.

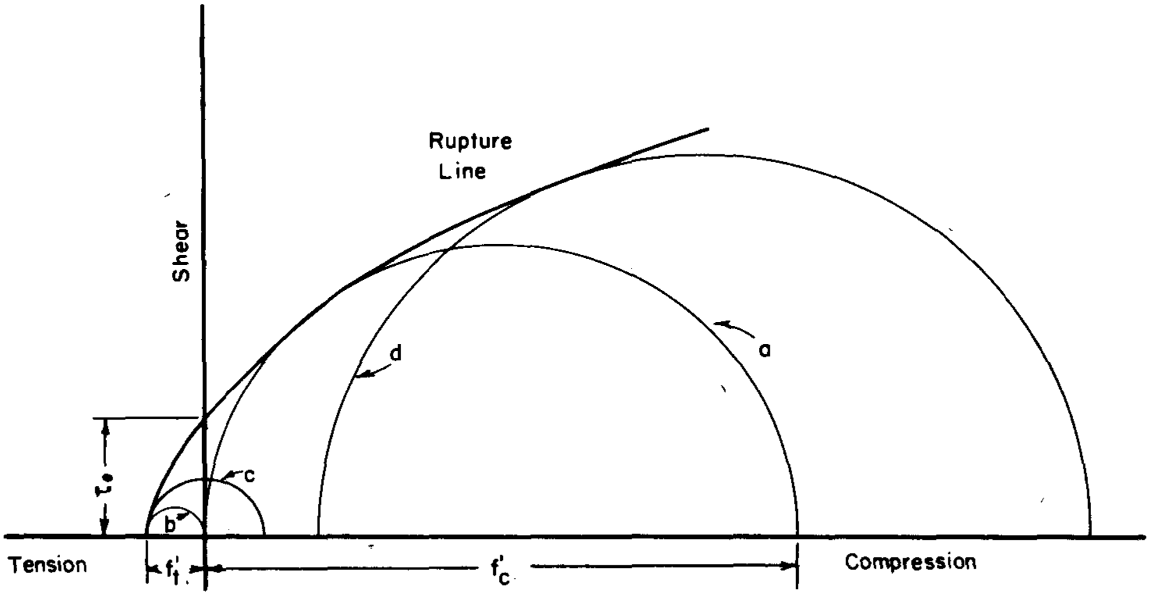

FIG. 3—Typical Mohr rupture diagram for concrete.

Since all of the circles shown are representative of the conditions at failure, the envelope curve shown on Fig. 3 is the rupture line. This line can be determined for a particular concrete only by making a series of tests with different combinations of stress, yielding a set of circles to which the rupture line must be tangent. The rupture line has been determined in this fashion in only relatively few cases. In the most extensive investigations, only tests under triaxial compression have been made. These tests yield results which are sufficient to define the rupture line only in the region to the right of the vertical axis. However, the rupture line has been extended to the vertical axis and beyond by statistical procedure [*37*] and by making use of the circle for the axial tension test (Circle *b*) [*39*]. The portion of the rupture line in the neighborhood of the vertical axis may also be determined from the results of tests under combined torsion and compression. At the present time, however, the strength characteristics of concrete under combined stresses are reasonably well known and understood only for conditions corresponding

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[3] For discussion of Mohr's circle and Mohr's rupture diagram, see paper by Richart, Brandizaeg, and Brown [*35*] p. 11.

to that portion of the rupture diagram to the right of the vertical axis.

The rupture line defines the limiting state of stress which can exist in the material before failure. If the combination and magnitudes of the stresses are such that the Mohr's circle touches the rupture line, failure will occur. For example, the standard cylinder tested in uniaxial compression will fail when the axial stress has been increased to such a value that Circle *a* touches the rupture line for the particular concrete being tested. The cylinder fails on a plane on which the normal and shearing stresses have the values given by the coordinates to the point at which the circle touches the rupture line. In the axial tension test, Circle *b*, only tensile stresses are present on the plane of failure. Similarly, in the torsion test, represented by Circle *c*, failure presumably occurs on a plane for which the conditions are similar to those in the tension test, since Circles *b* and *c* both touch the rupture line at the same point.

The strength of concrete in pure shear —that is, when no normal stresses are present on the plane of failure—is the stress τ_0 in Fig. 1, measured to the intersection of the rupture line with the vertical axis. On the basis of the available data, the value of the strength of concrete in pure shear is approximately 20 per cent of the compressive strength [*37–39*]. The shearing strength when normal stresses also occur on the plane of failure may be greater or less than τ_0, depending on whether the stresses are compression or tension.

The results represented by a circle such as *d* indicate that the axial compressive strength of concrete is appreciably increased by the existence of lateral confining pressures such as those exerted by the spiral reinforcement in a column or by that portion of column concrete surrounded by floor concrete. The increase in axial strength over the unconfined compressive strength ranges from as much as 4 to 5 times the value of the lateral stress for small confining pressures [*35,37*] to about 2.5 or 3 times that stress for large confining pressures [*37*].

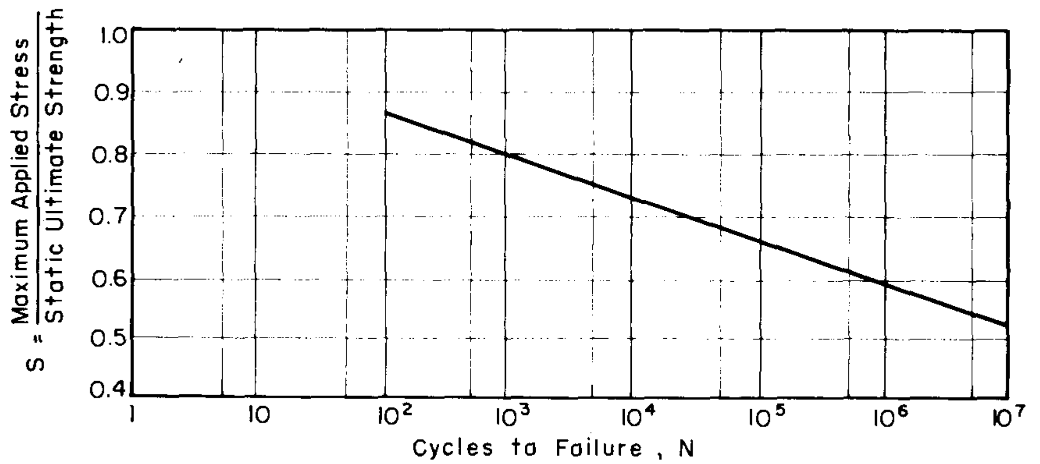

FIG. 4—Typical fatigue curve for concrete subject to repeated flexural load.

FATIGUE STRENGTH

Concrete will, when subjected to repeated load, fail at a load smaller than its static ultimate strength. Two excellent reviews of the behavior of concrete in fatigue were prepared by Nordby [*40*] and Murdock [*41*]. As may be seen from a typical fatigue curve for air-dried concrete in flexure, Fig. 4, the fatigue strength at 10 million cycles is about 55

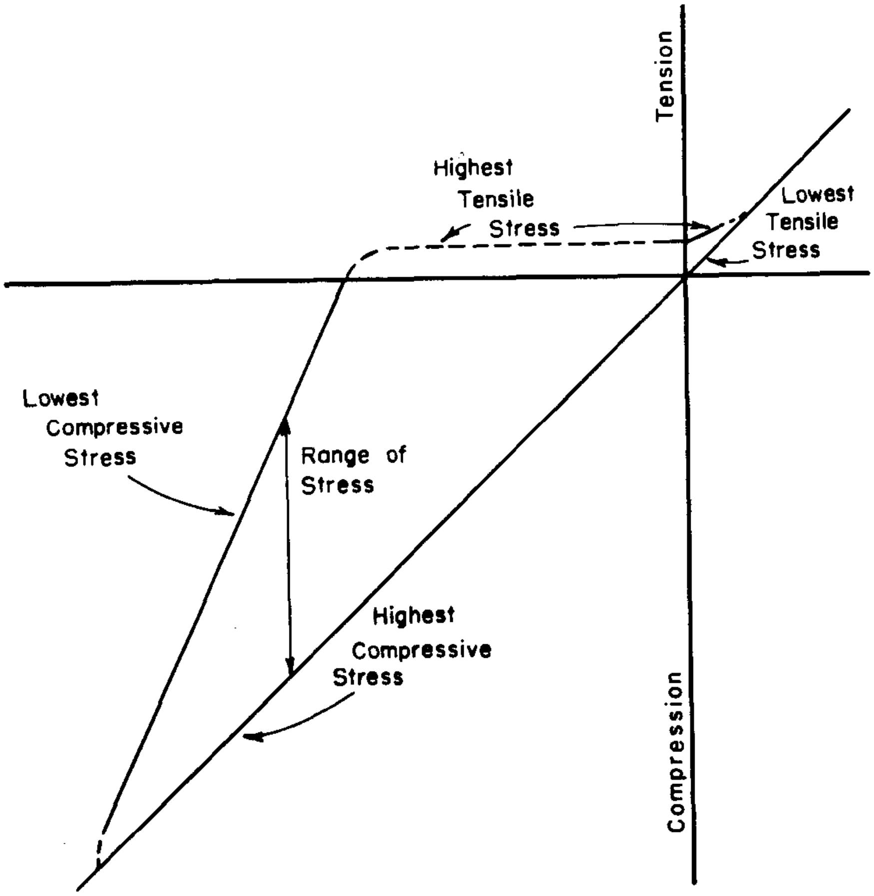

FIG. 5—Typical modified Goodman diagram.

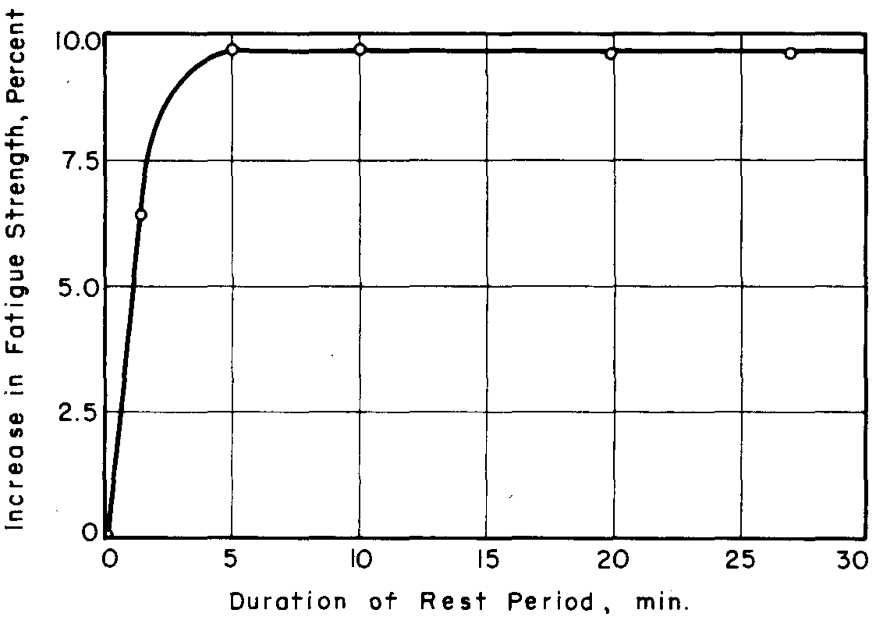

FIG. 6—Increase in fatigue strength as a result of rest periods.

per cent of the static flexural strength. The fatigue strength in axial compression is about 55 per cent of the static compressive strength. No data are available to establish the fatigue strength in axial tension, in shear, or under combined stresses. Concrete does not have a fatigue or endurance limit at least at less than 10 million cycles of load as most body-centered cubic metals do. Failure under repeated loads is especially important in pavement design.

Concrete is sensitive to the range of the repeated stress. This can best be described by a modified Goodman diagram, Fig. 5 [*41*,*42*]. Note that as the range of the repeated stress decreases the magnitude of the maximum load the specimen can carry increases. The curve also shows that whether a flexural specimen is loaded from zero to a maximum or in complete reversal the maximum tensile fatigue strength (in flexure) is the same. No data are available to establish the portion of the curve shown by the dashed line.

The frequency of loading in the range from 70 to 440 cpm has been found to have no effect on the fatigue strength [*43*].

Frequent rest periods during a fatigue test may raise the fatigue strength as much as 9 per cent higher than had there been no rest periods, Fig. 6. The fatigue strength increases as the rest periods are increased in duration to 5 min. No additional gain is obtained for longer rest periods [*41*].

Varying the magnitude of the maximum repeated load during a test affects the fatigue life of the concrete. However insufficient data are available to make any general conclusions. It is known, however, that the Miner hypothesis, which assumes linear damage accumulation, is not generally applicable to concrete [*41*].

Generally speaking, the various factors that affect static strength will affect fatigue in a similar manner. Thus, if fatigue response is expressed in terms of the static strength the fatigue response appears to be the same for all concrete.

Tests to determine the fatigue strength of concrete should be made on specimens as large as possible in order to decrease the influence of lack of homogeneity. The cross section of the specimen should be at least three times the maximum nominal size of the aggregate, and even larger dimensions might be desirable in some cases. Because of the large size of specimens used, the large testing machines required are usually capable of applying load at a rate ranging from only a few cycles a day to 500 cpm. Thus, it takes a minimum of two weeks to several months to apply as many as 10 million cycles of load. Because of the time involved, the specimens are generally aged and air-dried before being tested in order to prevent gain of strength during the test.

In general, the information on fatigue of concrete is meager. However, since field conditions are different than those of the conventional fatigue test in that mixed loading and rest periods usually occur as a matter of course, it appears safe to suggest that the fatigue life of concrete in the field is significantly greater than indicated by the conventional laboratory test without rest periods and at a constant maximum load.

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Hardened Concrete

ELASTIC PROPERTIES AND CREEP

By Robert E. Philleo[1]

When a load is applied to a body, the body is deformed. For a particular body loaded in a particular environment the amount of the deformation depends upon the magnitude of the load, the rate at which it is applied, and the elapsed time after the load application that the observation is made. Different materials vary widely in their response to load. This response is known as rheological behavior. While instantaneous effects and time-dependent effects are not entirely separable, it is common to consider them separately as elastic properties (instantaneous) and creep (time-dependent).

A knowledge of the rheological properties of concrete is necessary to compute deflections of structures, to compute loss of prestress in prestressed structures, to compute stresses from observed strains, and, when working stress design procedures are employed, to proportion sections and to determine the quantity of steel required in reinforced concrete members.

Although a vast amount of work has been done on the rheology of materials, much of it is not applicable to concrete. Because of the peculiar "gel" structure of cement paste, concrete behaves quite differently under applied load than does a crystalline material.

Elastic Properties

A body which returns to its original dimensions after enduring stress is elastic. A quantitative measure of elasticity is the ratio of stress to corresponding strain. Robert Hooke in 1678 discovered that for many materials this ratio is constant over a fairly wide range of stress. This ratio is termed the modulus of elasticity, and it has become one of the most commonly used parameters to describe material properties even though many materials do not exhibit a linear stress-strain relationship. Two additional terms are used to describe limits of elastic behavior: (1) proportional limit and (2) elastic limit. The proportional limit is defined in ASTM Definitions of Terms Relating to Methods of Mechanical Testing (E 6) as "the greatest stress which a material is capable of sustaining without any deviation from proportionality of stress to strain (Hooke's law)." The elastic limit is "the greatest stress which a material is capable of sustaining without any permanent strain remaining upon complete release of the stress."

Concrete has neither a definite proportional limit nor elastic limit. Therefore, the manner in which its modulus of elasticity is defined is somewhat arbitrary. Various forms of the modulus which have been used are illustrated on the stress-strain curve in Fig. 1. They

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are defined in ASTM Definitions E 6 as follows:

Initial Tangent Modulus—The slope of the stress-strain curve at the origin.

Tangent Modulus—The slope of the stress-strain curve at any specified stress or strain.

Secant Modulus—The slope of the secant drawn from the origin to any specified point on the stress-strain curve.

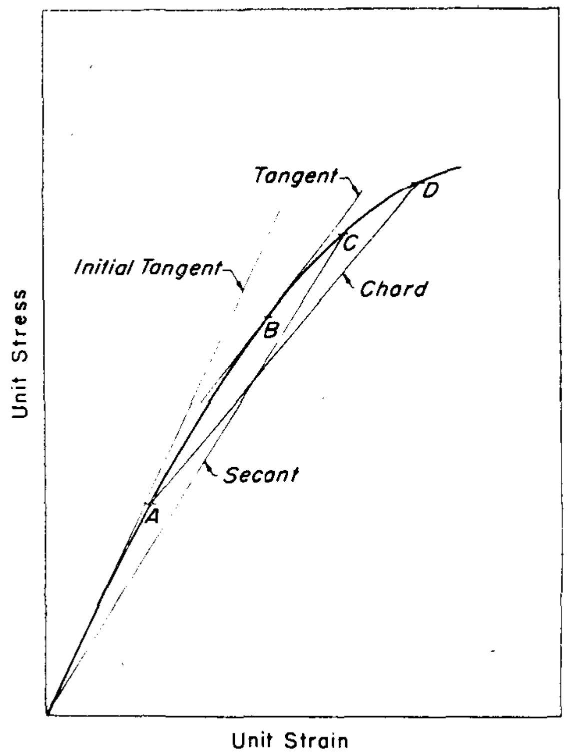

FIG. 1—Various forms of static modulus of elasticity.

Chord Modulus—The slope of the chord drawn between any two specified points on the stress-strain curve.

Modulus of elasticity may be measured in tension, compression, or shear. The modulus in tension is usually equal to the modulus in compression and is frequently referred to as Young's modulus of elasticity. The shear modulus, also called the modulus of rigidity or torsional modulus, is the ratio of shear stress to shear strain. Shear stress is defined in ASTM Definitions E 6 as "the stress or component of stress acting tangential to a plane," and shear strain is defined as "the tangent of the angular change between two lines originally perpendicular to each other."

When stress is applied in a given direction, there are changes in dimension in directions perpendicular to the direction of the applied stress, as well as in the direction of the stress. The magnitudes of the lateral strains are different for different materials. Thus, two parameters are required to describe the elastic behavior of a material. The parameters may take many forms, but the two most commonly used are Young's modulus of elasticity and Poisson's ratio. Poisson's ratio is defined in ASTM Definitions E 6 as "the absolute value of the ratio of transverse strain to the corresponding axial strain resulting from uniformly distributed axial stress below the proportional limit of the material." The transverse strains are opposite in direction to the axial strains. For a material obeying Hooke's law, Poisson's ratio is constant below the proportional limit. It can be shown that the following relationship exists among Young's modulus of elasticity, modulus of elasticity in shear, and Poisson's ratio:

$$\mu = \frac{E}{2G} - 1$$

where:

μ = Poisson's ratio,
E = Young's modulus of elasticity, and
G = modulus of elasticity in shear.

Thus, if any two of these quantities are determined, the third can be calculated.

It can be shown that the natural frequency of vibration of an elastic body is proportional to the square root of either Young's modulus or the shear modulus, depending on the mode of vibration. Likewise, the velocity with which a compressional shock wave travels through an elastic body is proportional to the square root of Young's modulus. Since these phenomena and their application to concrete are discussed elsewhere in this volume, only the measurements of modulus of elasticity by "static" test methods will be described here.

FIG. 2—Compressometer.

Modulus of Elasticity in Compression:

Since structural concrete is designed principally for compressive stresses, by far the greatest amount of work on the elastic properties of concrete has been done on concrete in compression. The only ASTM standard method of test for static modulus of elasticity of concrete, ASTM Test for Static Young's Modulus of Elasticity and Poisson's Ratio in Compression of Cylindrical Concrete Specimens (C 469), is a compressive test method. It stipulates a chord modulus between two points on the stress strain curve defined as follows: the lower point corresponds to a strain of 50 μ in./in., and the upper point corresponds to a stress equal to 40 per cent of the strength of concrete at the time of loading. The lower point is near the origin but far enough removed from the origin to be free of possible irregularities in strain readings caused by seating of the testing machine platens and strain measuring devices. The upper point is taken near the upper end of the working stress range assumed in design. Thus, the determined modulus is approximately the average modulus of elasticity in compression throughout the working stress range.

The 6 by 12-in. cylinder is the specimen size most commonly used for the determination of the modulus of elasticity in compression. In order to compensate for the effect of eccentric loading or nonuniform response by the specimen, strains should be measured along the axis of the specimen or along two or more gage lines uniformly spaced around the periphery of the cylinder. The selection of the gage length is important. It must be large in comparison with the maximum aggregate size so that local strain discontinuities do not unduly influence the results, and it must be large enough to span an adequate sample of the material. It must not, however, encroach on the ends of the specimen. Because of restraint where the specimen is in contact with the steel platens of the testing machine, strains near the ends may differ somewhat from strains elsewhere in the specimen. ASTM Method C 469 specifies that the gage length shall be not less than three times the maximum size of aggregate nor more than two thirds the height of the specimen. Half the specimen height is said to be the preferred gage length. A convenient device for measuring the strains is a compressometer, such as the one pictured in Fig. 2. The lower yoke is rigidly attached to the specimen, whereas the upper yoke is free to rotate as the specimen shortens. The pivot rod and dial gage are arranged so that twice the average shortening of the specimen is read on the dial. This type of device was used in the first comprehensive investigation of modulus of elasticity by Walker [*1*],[2] and it is cited in ASTM Method C 469 as an acceptable device. Electrical strain gages have also been used successfully. These include both gages embedded along the axis of the specimens and those bonded to the surface. There are also available compressometers in which the strain, instead of being observed on a dial gage, is indicated and recorded by an electrical device such as a linear differential transformer.

Because the test is intended to measure only time-independent strains, it is important that the specimen be loaded expeditiously and without interruption. For this purpose an automatic stress-strain recorder is helpful but not essential. Since it is desired that only those length changes due to load be measured, temperature and moisture conditions should be controlled during the test.

A large numer of results have been reported in the literature [*2–4*]. The range of results has been from 1,000,000 to 3,000,000 psi for structural lightweight concrete and from 2,000,000 to 5,000,000 psi for normal weight concrete. A simple relationship between modulus of elasticity and other easily-measured properties of concrete, such as strength and unit weight, would be useful. While no theoretical relationship exists, an approximate equation adopted by the American Concrete Inst. (ACI) Building Code [*5*], which is discussed later, has practical value.

Although the standard method of test is not concerned with the behavior of concrete at stresses above 40 per

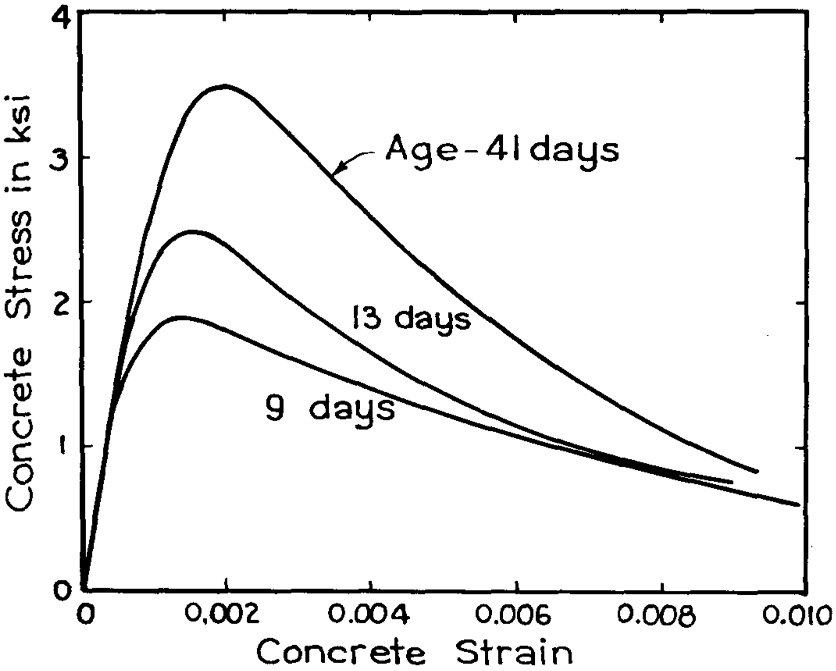

FIG. 3—Complete stress-strain curves (taken from Ref. *15*).

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

cent of the ultimate strength, the shape of the stress-strain curve at high stresses is of significance in determining the ultimate load-carrying capacity of a concrete member. In most testing machines concrete cylinders, except those with very low strength, fail suddenly shortly after the maximum load has been attained. It has been demonstrated [*6–10*, *15*] that such failures are related to the properties of the testing machine rather than the properties of the concrete. By using a suitably stiff testing machine or by artificially stiffening a testing machine by surrounding the concrete specimen with steel springs, it is possible to obtain stress-strain curves covering a strain range several times as great as that required to attain maximum stress. Fig. 3 illustrates such stress-strain curves.

Modulus of Elasticity in Tension:

A limited amount of work has been done on the determination of Young's modulus in tension of concrete [*11–13*]. The test is complicated by the limited stress range of concrete in tension and the problems associated with gripping the tension test specimens. Recent work has been concerned with the development of specimen shapes which will insure uniform stress distribution throughout the section in which measurements are made. Since Young's modulus in tension does not appear to differ from Young's modulus in compression at low stresses and since there is no strain range beyond maximum tensile stress to be investigated, there is relatively little stimulus for the development of data on the tensile modulus.

Modulus of Elasticity in Flexure:

Since a principal use of reinforced concrete is in flexural members, several investigators have determined Young's modulus on specimens loaded as beams. An obvious approach is to measure deflections caused by known loads and to calculate the modulus of elasticity from well-known beam deflection formulas. Unfortunately, the depth-to-span ratios of concrete beams normally used for such tests are so large that shear deflection comprises a significant part of the total deflection. In applying shear corrections, certain other corrections must be made to take care of discontinuities in the shear deflection curves at load points. The corrections most commonly used are those of Seewald [*14*]. For center-point loading he gives the following deflection formula:

$$\delta = \frac{Pl^3}{48EI}\left[1 + (2.4 + 1.5\mu)\left(\frac{h}{l}\right)^2 - 0.84\left(\frac{h}{l}\right)^3\right]$$

where:

δ = maximum deflection,

P = applied central load,

l = distance between supports,

E = modulus of elasticity,

I = moment of inertia of the section with respect to the centroidal section,

μ = Poisson's ratio, and

h = depth of the beam.

The portion of the expression outside the brackets is the simple beam formula.

In some tests, strain gages have been placed on the tensile and compressive faces to determine strain as a function of applied load. The load may be converted to fiber stress by standard beam formulas. For such tests the beam is usually loaded at two symmetrical points so that there is a constant stress condition between the two load points. Such tests have the advantage of indicating the position of the neutral axis, but if the stress-strain curve is nonlinear the computed stresses and, therefore, the computed modulus of elasticity are in error.

In a comprehensive investigation of the compression side of a concrete beam Hognestad, Hanson, and McHenry [*15*] utilized test specimens of the types shown in Fig. 4. The central prismatic portion of the specimen is loaded both concentrically and eccentrically. The eccentric load is continuously adjusted so that the strain on one surface remains zero. Thus, the section simulates that portion of a beam between the neutral axis and the extreme compressive fiber. The complete stress-strain curve in flexure can be determined from the observed loads and strains if the following two assumptions are made:

1. The distribution of strain across the section is linear.

2. All fibers follow the same stress-strain curve. The first has been amply verified in these and other tests. The second depends on the speed of testing. If the specimen is loaded expeditiously, time-dependent effects are held to a minimum. In these tests the stress-strain curves in flexure agreed very well with stress-strain curves obtained on companion cylinders concentrically loaded in compression both below and above the maximum stress.

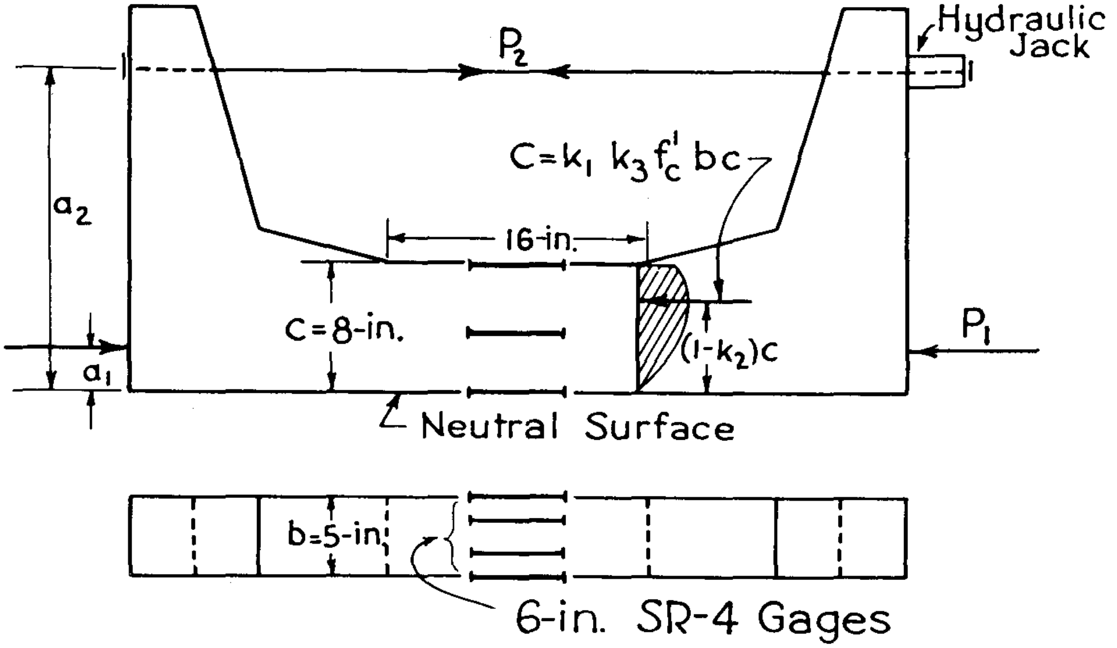

FIG. 4—Test specimens for stress distribution in a beam (taken from Ref. *15*).

Modulus of Elasticity in Shear:

The shear modulus, or modulus of rigidity, is most often determined dynamically or by calculation from Young's modulus and Poisson's ratio. When a direct static determination is desired, a torsion test is the common procedure. When a given torque is applied to a body the angle of twist in a given length is inversely proportional to the shear modulus as indicated by the following equation:

$$G = \frac{L}{\phi I}$$

where:

G = modulus of elasticity in shear,
L = torque,
ϕ = angle of twist per unit length, and

I = polar moment of inertia of the cross section.

A method for testing concrete cylinders has been described by Andersen [*16*]. He used a level bar with micrometer adjustment for measuring differences in angular change of two radial arms placed a fixed distance apart on a horizontally positioned specimen.

Poisson's Ratio:

Static determinations of Poisson's ratio are made by adding a third yoke and second dial gage to a compressometer so that a magnified transverse strain may be measured, as well as a magnified axial strain or by mounting strain gages on the surface of a specimen perpendicular to the direction of loading. The same considerations apply to gage length for lateral strain measurement as for longitudinal strain measurement except that for lateral strains there is no upper limit since end restraint is not a factor. Procedures for determination of Poisson's ratio are included in ASTM Method C 469. Poisson's ratio is also commonly computed from results of Young's modulus and shear modulus determined dynamically. The static value at stresses below 40 per cent of the ultimate strength is essentially constant; for most concretes the values fall between 0.15 and 0.20. The dynamic values are usually in the vicinity of 0.25. At high stresses or under conditions of rapidly alternating loads a different picture emerges. Probst [*17*] has shown a systematic increase in the value of Poisson's ratio with stress repetition and Brandtzaeg [*18*] has shown a marked increase at very high stresses. When the value is below 0.50 there is a decrease in volume of the body as a compressive load is applied. Brandtzaeg's work indicates that above about 80 per cent of the strength there is an increase in volume as additional compressive loads are applied.

CREEP

Creep is defined in ASTM Definitions E 6 as "the time-dependent part of strain resulting from stress." All materials undergo creep under some conditions of loading. In metals and other crystalline materials creep has been attributed to slip in crystals. While slip of this nature undoubtedly occurs in aggregate particles and within crystalline particles that are part of hydrated paste, there is ample evidence that these are only secondary factors in the creep of concrete. Crystalline slip is normally detectable only above some threshold level of stress. Creep of concrete is observed at all stresses. Furthermore, creep of concrete is approximately a linear function of stress up to 35 to 40 per cent of its strength. This behavior is not associated with crystalline slip. Finally, the order of magnitude of concrete creep is much greater than that of crystalline materials except for metals in the final stage of yielding prior to failure. Thus, concrete creep is considered to be an isolated rheological phenomenon associated with the gel structure of cement paste. This unique aspect of the problem has the advantage that creep may be measured without extremely sensitive equipment but the disadvantage that little of the research which has been done on other materials is applicable to concrete. A review of current knowledge of creep is to be found in the 1964 ACI *Symposium on Creep of Concrete* [*19*].

The particular aspect of the gel structure of concrete which causes its unusual behavior is the accessibility of its large internal surface to water. In fact, Mullen and Dolch [*20*] found no creep at all in oven-dried pastes. The movement of water into and out of the gel in response to changes in ambient humidity produces the well known shrinking and swelling behavior of concrete. A principal view among investigators [*21–25*] is that creep is closely related to shrinkage. In creep, gel water movement is caused by changes in applied pressure instead of differential hygrometric conditions between the concrete and its environment. This concept is supported by the similar manner in which creep and shrinkage curves are affected by such factors as water-cement ratio, mix proportions, properties of aggregate, compaction, curing conditions, and degree of hydration.

Another explanation of the effect of gel water [*26,27*] is delayed elasticity. If a load is suddenly imposed on a body consisting of a solid elastic skeleton with its voids filled with a viscous fluid, the load will be carried initially by the fluid and will gradually be transferred to the skeleton as the fluid flows under load. This is the behavior exhibited by the rheological model known as a Kelvin body, which consists of a spring and dashpot in parallel. The concept of delayed elasticity has been chiefly responsible for the widespread attempts to reproduce the rheological behavior of concrete by means of rheological models.

Creep of concrete has been attributed by some [*28–31*] to viscous flow of the cement paste. The reduction in strain rate with time has been attributed by these investigators both to the increasing viscosity of the paste and to the gradual transfer of load from the cement paste to the aggregate. This concept is supported by the concept that creep strain is proportional to the applied stress over a wide range of stress. A convincing argument against it is the fact that the volume of concrete does not remain constant while it creeps. In fact Poisson's ratio for creep has usually been found to be less than for elastic stress. In at least one investigation [*32*] Poisson's ratio was found to be zero. Another argument against the concept is the partial recovery of creep when the load is removed.

The fact that creep is associated primarily with the cement paste phase of concrete produces a serious difficulty in the interpretation and application of creep data. Unless the work is restricted to very mature concrete, the specimens do not maintain constant physical properties throughout the test. Creep measurements must necessarily be made over a considerable period of time and during that time the cement paste continues to hydrate. Frequently information is desired at early ages when the cement is hydrating relatively rapidly.

Measurement of Creep:

The use to be made of creep measurements usually determines the age at which creep tests are begun and the stress level to which specimens are loaded. A test procedure has been standardized in ASTM Method of Test for Creep of Concrete in Compression (C 512 – 64 T). The method stipulates loading moist-cured specimens at an age of 28 days to a stress not exceeding 40 per cent of the strength of the concrete at the time of loading, although provision is made for other storage conditions or other ages of loading. The stress is restricted to the range throughout which creep has been found to be proportional to stress. Limitations on gage lengths similar to those in the test for modulus of elasticity apply. The age of loading for a standard test is necessarily arbitrary. The method is intended to compare the creep potential of various concretes. Testing at a single age of loading is satisfactory for this purpose. It is required in the test method that the stress remain constant throughout the one-year duration of the test within close tolerances. The load may be applied by a controlled hydraulic system or by springs, provided in the latter case the load is measured and adjusted frequently. It is also required that there be companion unloaded specimens. Length changes of these specimens are measured and subtracted from the length changes of the loaded specimens to determine creep due to load. This correction is intended to eliminate the effects of shrinkage and other autogenous volume change. While this correction is qualitatively correct and yields usable results, most modern theories deny the independence of shrinkage and creep and thus indicate that the two effects are not additive as assumed in the test. As more is learned about these phenomena, this test detail will probably be altered.

As with testing for modulus of elasticity, most creep testing has been concerned with specimens subjected to compression. Several investigators [*11*, *33–35*] have studied creep in flexure because of the obvious application to beam deflections. A limited amount of work has been done in tension [*32*,*36*,*37*] and in torsion [*16*,*38*,*39*].

Creep Equations:

Creep under constant stress always proceeds in the same direction at a rate which is a decreasing function of time. It is, therefore, common to plot creep test results on a semilogarithmic graph in which the linear axis represents creep strain and the logarithmic axis represents time. Many sets of data show an approximately straight line over a considerable period of time. This has led to the development of numerous logarithmic equations for creep. Such an equation is cited in ASTM Method C 512. In that test method the results are reported by listing the strains at specified ages up to a year. In addition it is suggested that the report contain the parameters of the following equation:

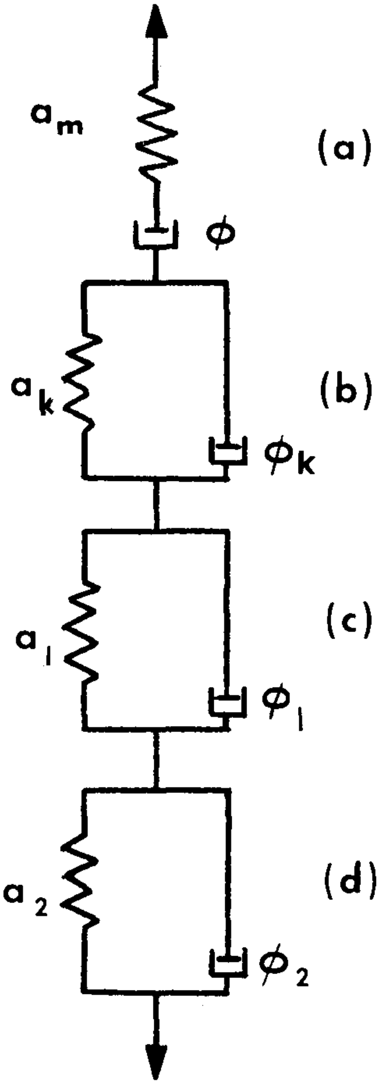

FIG. 5—Typical rheological model for representing creep (taken from Ref. *41*).

$$\epsilon = \frac{1}{E} + F(K) \log_e (t + 1)$$

where:

ϵ = total strain per psi,

E = instantaneous elastic modulus, in psi,

$F(K)$ = creep rate, calculated as the slope of a straight line representing the creep curve on the semilog plot, and

t = time after loading, in days.

While it is not intended that a theoretical logarithmic law should be inferred from the equation, the slope of the least squares line is a convenient parameter for comparing the creep characteristics of different concretes.

Most of the other equations which have been used to describe creep have been based on the assumption that there is a limiting value of creep. Typical of these is the Lorman [*24*] equation:

$$c = \frac{mt}{n + t}\sigma$$

where:

c = creep strain after time, t, for a sustained stress, σ,
m = ultimate creep strain per unit of stress, and
n = the time at which half the ultimate creep is attained.

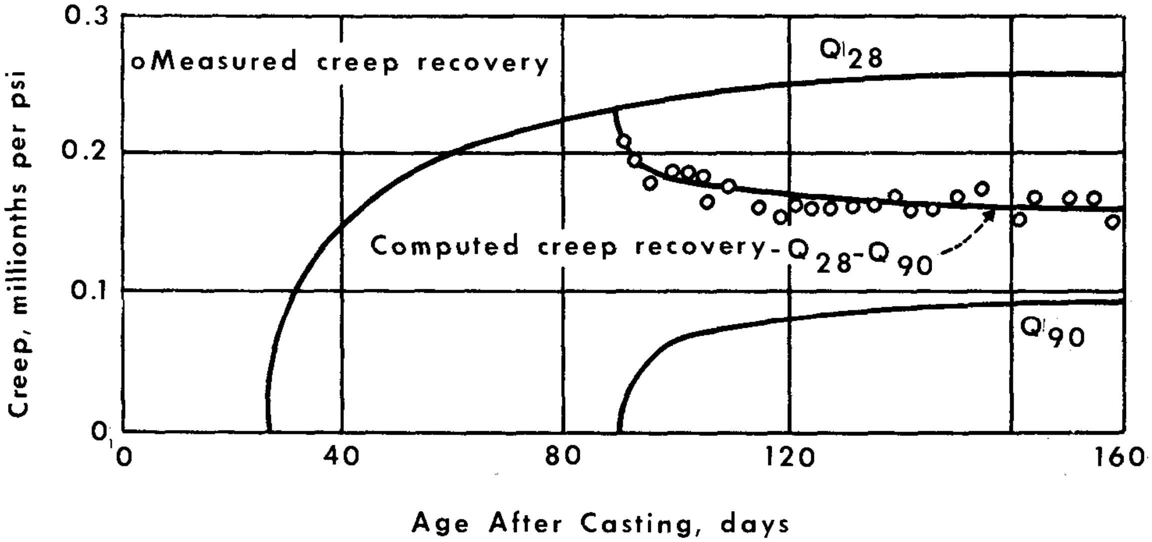

FIG. 6—Principle of superposition (taken from Ref. *40*).

McHenry [*40*] added coefficients to take into account the effects of hydration during the loading period with the following triple exponential equation:

$$\epsilon_c = \alpha(1 - e^{-rt}) + \beta e^{-pk}(1 - e^{-mt})$$

where:

ϵ_c = specific creep,
k = the age at time of loading,
t = time after loading, and
α, β, r, p, and m are empirical constants.

Rheological Models:

Many investigators have used mechanical models as an aid in setting up mathematical equations. The elements normally used are the ideal spring, in which force is proportional to strain, and the ideal dashpot, in which force is proportional to the rate of strain. They are usually grouped together in pairs. A spring and dashpot in parallel form a Kelvin unit, while a spring and dashpot in series form a Maxwell unit. By selecting an appropriate group of elements it is possible to produce a system empirically which can reproduce any given set of creep data. The models are frequently simplified somewhat if some of the spring or dashpot constants are made time-dependent or stress-dependent. While the model is an aid in visualizing creep behavior and in writing equations for that behavior, it should not be considered as representing the structure of the real material. Typical of models which have been proposed is that of Freudenthal and Roll [*41*], which is pictured in Fig. 5.

Principle of Superposition:

Of considerable practical value is a knowledge of the response of concrete to changing loads. While many of the investigations have included complete unloading as a portion of the study, there has been only a limited study of other forms of variable loading. McHenry [*40*] proposed a theory of superposition whereby the effect of each change in load is assumed to be added algebraically to the effects of all previous loads. The material is assumed to have a perfect memory. The effect of each increment of load lasts forever. Figure 6 shows how the theory applies to creep recovery. Three sets of specimens are required to produce such a figure. Two are loaded at an age of 28 days. One of these is unloaded at 90 days. A third set is loaded at 90 days to the same stress level as the original sets. The principle of superposition is valid if the strain in the last set equals the difference in strains of the first two sets. The principle is consistent with the preponderance of observations that only partial recovery occurs when a specimen is unloaded. While there is not unanimous agreement [*42*] on the principle of superposition, it is used regularly in computing stresses in mass concrete from measured strains.

Effect of Paste Content:

Creep is influenced by mix proportions, type of aggregate, conditions of storage, and age at loading. Most of the creep studies which have been conducted have been for the purpose of determining the effect of one or more of these variables on creep. One aspect of the effect of mix proportions is of continual interest in the field of mass concrete. That is the relationship between creep and paste content. Mass concrete commonly contains 6-in. aggregate. Fabrication and testing of concrete specimens containing such large aggregate is expensive. Work at the Waterways Experiment Station of the Corps of Engineers [*43*] and at the University of California [*44*] has demonstrated that the creep of sealed specimens, having identical water-cement ratios and air contents in the mortar phase but different amounts and sizes of a given coarse aggregate, is proportional to the paste content. This finding has made it possible to develop significant data for mass concrete from small specimens.

Effect of Specimen Size:

It has been demonstrated [*43,45*] that creep of sealed specimens is independent of specimen size. This observation plus the observation concerning mass concrete in the preceding paragraph indicate that the techniques and specimens of ASTM Method C 512 are applicable to all types of concrete sealed to prevent loss of moisture. For unsealed specimens exposed to a drying atmosphere it is evident that there must be a size effect associated with the moisture gradients within the specimen. The creep of a structure may be only a fraction of that in a test specimen. Ross [*46*] investigated the effect of specimen size on shrinkage and found at each age an excellent correlation between shrinkage and the ratio of exposed surface to volume of specimen independent of the shape of the specimen. In recent unpublished work at the Portland Cement Assn. the effect of size on both shrinkage and creep was investigated, and both were found to be dependent only on the ratio of surface to volume. Information of this sort may make it possible to apply correction factors to the data obtained from ASTM Method C 512 to determine the creep in any size and shape of structure.

SIGNIFICANCE OF PROPERTIES

Deflection of Flexural Members:

A footnote in the ACI Building Code [5] states: "Deflections in reinforced concrete structures depend on the elastic and inelastic properties of concrete and steel, as well as on shrinkage and creep which, in turn, are influenced by temperature and humidity, curing conditions, age of concrete at the time of loading, and other factors." It is not uncommon for a reinforced concrete flexural member eventually to reach a deflection three times as great as its initial deflection. While a precise prediction of these deflections is possible only if the elastic and creep properties are known, the required precision is usually not great. Relatively little creep testing is directed to predicting deflections of specific structures.

Loss of Prestress:

In contrast to the lack of precision needed for deflection measurements, an accurate knowledge of the early-age rheological properties of concrete is valuable to the prestressed concrete industry. After the prestress is applied, there is a loss of prestress resulting from creep of the concrete, shrinkage of the concrete, and relaxation of the steel. Since the initial prestress is limited by the strength of the steel and the load-carrying capacity of the member is limited by the residual prestress, a knowledge of the factors governing loss

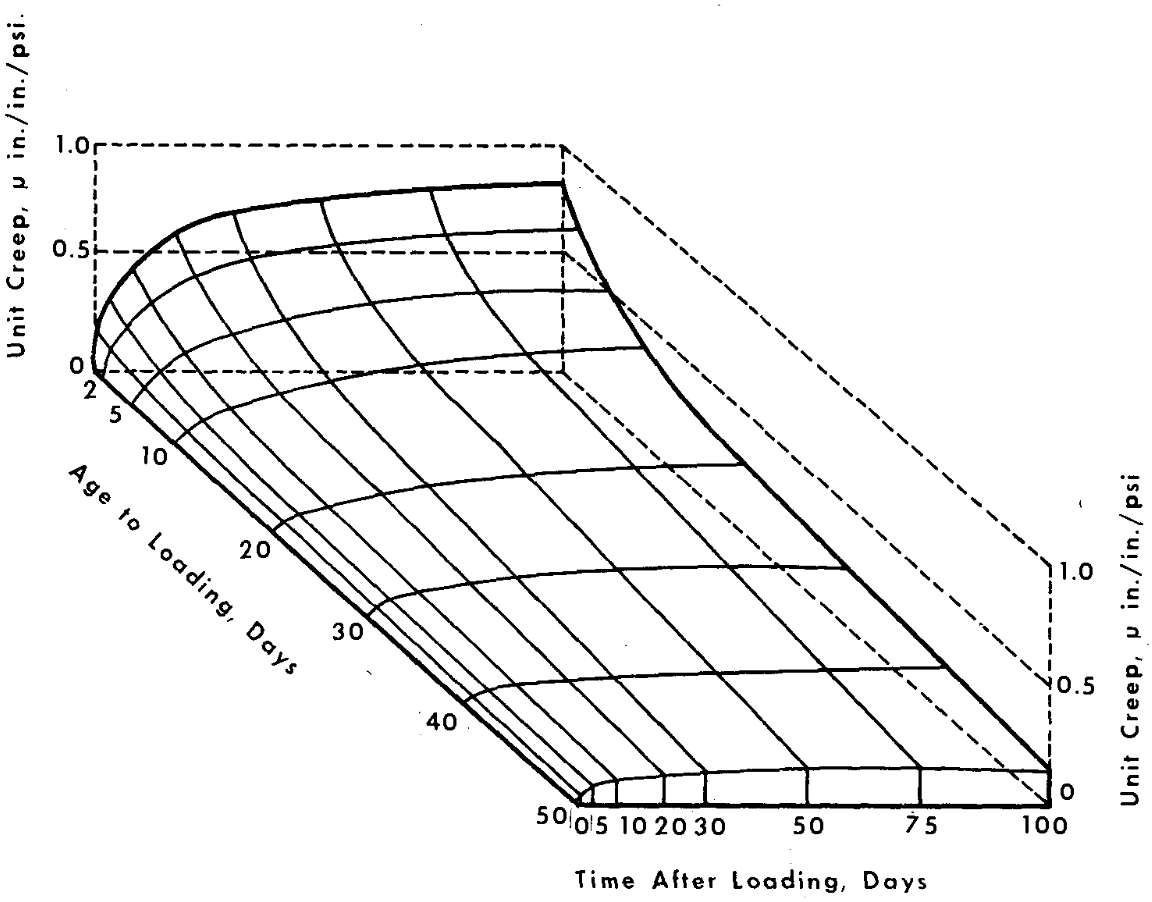

FIG. 7—Typical creep surface.

of prestress has important economic implications. A substantial amount of work has been done in this field recently.

Structural Design:

When reinforced concrete is designed by working stress theory, perfect bond between the concrete and steel is assumed under design load conditions. Therefore, the load carried by the steel is a function of the ratio of the modulus of elasticity of steel to that of concrete. Design is impossible unless these quantities are known or assumed. For many types of structures such as arches, tunnels, tanks, and flat slabs, a knowledge of Poisson's ratio as well as modulus of elasticity is needed.

As discussed previously, a knowledge of the complete stress-strain curve for concrete has assisted in the refinement of ultimate strength design theory.

Stress Calculations:

A prerequisite for the calculation of stresses from measured strains is a complete knowledge of a material's rheological behavior. When stresses result from nonuniform temperature or humidity as well as from applied load, the thermal coefficient of expansion and shrinkage data must also be available. Perhaps the most extensive use of creep data has been in the stress analysis of mass concrete structures [*47*]. These structures are unreinforced and contain complicated stress distributions because of temperature conditions resulting from the heat of hydration of cement. The procedure requires the installation of a large number of strain meters within the structure during construction. It is necessary to run creep tests on sealed specimens in the laboratory at enough ages of loading so that by interpolation and extrapolation a complete knowledge of rheological behavior is available. Such data can be represented pictorially by a creep surface such as that shown in Fig. 7. To convert strain data to stress, it is assumed that creep is a linear function of stress and that the principle of superposition applies. At the location of each strain meter, stress calculations must be made continually from the time the concrete hardens to the end of the period for which results are desired. The time is divided into small intervals. If during any interval the change in strain is different from that which would be expected from the creep produced by the sum of all the stresses present at the point at the start of the interval, the change in strain must be accounted for by adding or subtracting an amount of stress sufficient to produce the desired strain increment at the appropriate age of loading. By this technique it has been possible to obtain an accurate assessment of the safety of dams and to evaluate the effectiveness of various methods of temperature control. A discussion of the accuracy of the method has been given by Pirtz and Carlson [*48*].

SPECIFICATIONS

Limits on rheological properties are almost never included in specifications. In some structures in which designers wished to minimize deflections, contractors have been restricted to those aggregates among the economically available materials which have been demonstrated to produce concrete having the lowest creep.

It has been pointed out that a knowledge of modulus of elasticity is required for some aspects of structural design, and where building codes impose limits on deflections a knowledge of creep behavior is also necessary. Specifications for structural concrete are primarily concerned with strength. It has not been considered feasible to complicate the specifications by additional rheological requirements. The problem has been approached in the ACI Building Code [5] by assuming that the modulus of elasticity is related to strength and unit weight as follows:

$$E = W^{1.5}\,33\,\sqrt{f'c}$$

where:

E = modulus of elasticity, in psi,
W = unit weight, in pounds per cubic foot, and
$f'c$ = specified compressive strength, in psi.

The ratio of long-time deflection to immediate deflection is assumed to vary from 1.8, when the amount of compression reinforcement equals the tension requirement, to 3.0, when there is no compression reinforcement. Neither of these estimates can claim a high degree of precision; both are probably adequate for the intended purpose. However, as structural design increases in sophistication and as applications of prestressed concrete increase, the interest in rheological properties is increasing. This interest is evident in the steadily increasing amount of creep testing being performed.

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Hardened Concrete

DYNAMIC TESTS

E. A. WHITEHURST[1]

For many years one of the goals of those engaged in the control of concrete quality and in the service behavior of concrete has been the development of suitable nondestructive tests to supply the information desired. To be of greatest usefulness, such tests should be applicable to concrete in the structure. It will be seen that the dynamic tests included in the following discussion, despite present limitations in application and in interpretation, are bringing the desired goal within reach in many respects.

For the purpose of this discussion, dynamic testing will be defined as that in which the load is applied and removed in a manner such that the effects of creep during testing are negligible, and which does not usually result in destruction or damage to the concrete. In general, it is found that values of Young's modulus of elasticity computed from dynamic tests are somewhat higher than those determined for slower applications of load in which both elastic and plastic deformations may occur.

Tests complying with this definition and in sufficiently wide use to warrant consideration may be subdivided into three groups:

1. Those identified as sonic tests, generally involving determination of the resonant frequency of a specimen.
2. Those identified as pulse velocity tests, generally involving measurement of the velocity of a compressional pulse travelling through the concrete.
3. Those involving the measurement of rebound distance of a hammer after striking a blow of controlled intensity, or the diameter of indentation caused by such a blow.

SONIC TESTS

The expression "sonic testing" is generally considered to include all testing of concrete which involves the generation of a sustained vibration in the concrete. By far the majority of such tests involves the determination of the fundamental resonant frequency of a specimen.

Equipment used to perform tests of this nature varies from the hammer and home-made sonometer reported by Powers [*1*][2] to electronic signal generators for driving the specimen and highly complex electronic counters for measuring the actual number of vibrations per unit of time. The hammer-sonometer method has been largely superseded by other methods, primarily because of the difficulty encountered by many operators in matching the tone emitted by the concrete specimen to the tone of the sonometer and because, in some cases, the striking of the specimen resulted in a change in its characteristics [*2*]. The use of the complex counter is generally restricted to research laboratories. The

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

large bulk of the apparatus falls between these extremes.

The components required for performing a test of this nature are an audio signal generator, an amplifier, a driving unit, a pickup unit, another amplifier, and a metering device. These are available individually from a number of sources and are also available in a combined form suitable for direct use in testing of this nature. The driving unit is frequently a permanent-magnet speaker with a rod attached to the speaker coil. The pickup is usually a piezoelectric crystal, often a phonograph pickup cartridge.

In earlier tests, the specimen was supported on knife-edges located at the nodal points for flexural vibration (a distance of 0.224 of the specimen length from each end). The driver was placed in the center of the specimen and the pickup at one end. Sufficient power was applied to the driver to cause mild vibration of the specimen. The frequency of the oscillator was then varied. When the frequency approached that of resonance for the specimen, the amplitude of specimen vibration increased considerably. The oscillator was tuned until a maximum indication was observed on the metering device. The frequency at which this occurred was recorded as the fundamental transverse frequency of the specimen.

Subsequent investigations indicated that if the specimen was allowed to rest uniformly on a sheet of soft sponge rubber the restraint on the specimen would be sufficiently low to remove the necessity for mounting it at its nodal points. This has now become a generally accepted practice. Considerable attention has also been directed to the use of torsional vibration for testing concrete specimens. In this technique, generally applied to prisms, the driving unit is placed against one corner of the specimen and the pickup against a diagonally opposite corner. The specimen is thus vibrated with a twisting motion, the node occurring at the center of the beam. Further investigations have included the use of longitudinal vibrations in which the driving unit is placed against one end of the specimen and the pickup unit against the other. In this case, also, the nodal point occurs at the center of the specimen.

ASTM Test for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens (C 215) makes provisions for testing concrete specimens for fundamental transverse, longitudinal, and torsional frequencies, and provides a schematic diagram illustrating use of the components enumerated above. The relationships between the several fundamental frequencies and other properties of the concrete are given as follows:

$$\text{Dynamic } E = CWn^2 \ldots\ldots (1)$$

$$\text{Dynamic } E = DW(n')^2 \ldots\ldots (2)$$

$$\text{Dynamic } G = BW(n'')^2 \ldots\ldots (3)$$

where:

Dynamic E = dynamic Young's modulus of elasticity, psi,
Dynamic G = dynamic modulus of rigidity, psi,
W = weight of specimen, lb,
n = fundamental transverse frequency, cps,
n' = fundamental longitudinal frequency, cps, and
n'' = fundamental torsional frequency, cps.

The constants C, D, and B are factors depending for their values upon the shape of the specimen tested and upon Poisson's ratio. Detailed instructions for their calculation may be found in ASTM Method C 215.

Where E and G are determined as

outlined above, Poisson's ratio may be calculated from the relationship:

$$\mu = \frac{E}{2G} - 1 \qquad (4)$$

Several investigators [*3*,*4*] have shown that a somewhat different characteristic of concrete, its damping capacity, may be determined from studying the behavior of a specimen vibrating at or near resonance. Two measures of this characteristic are suggested, the damping constant and the logarithmic decrement of a free vibration.

The damping constant is given by:

$$Q = \frac{f_0}{f_1 - f_2} \qquad (5)$$

where:

f_0 = resonant frequency of vibration, cps, and
f_1, f_2 = frequencies on either side of resonance at which the amplitude is $1/\sqrt{2}$ times the amplitude at resonance.

These values may be determined if a meter is used as the indicating device for determining resonance, provided that a sufficiently precise method of determining vibration frequency is available. This becomes extremely important since the frequency range between f_1 and f_2 is very small indeed. A frequency counter has been found to be satisfactory for this type of work.

The logarithmic decrement is given by:

$$\delta = \ln \frac{A_1}{A_2} \qquad (6)$$

where:

δ = logarithmic decrement, and
A_1, A_2 = amplitudes of two successive vibrations after the driving force has been removed from the specimen.

To determine A_1 and A_2, a cathode-ray oscillograph may be used as the indicator. After resonance has been located, the driving oscillator is turned off and the decay of the specimen vibration recorded on a moving film strip. When the film has been developed, the amplitudes of successive cycles may be accurately measured.

The damping constant and logarithmic decrement are related by:

$$Q = \frac{\pi}{\delta} \qquad (7)$$

Both of the above methods have a disadvantage in that the damping effects of the specimen supports must be extremely low. In an effort to minimize these effects, Obert and Duvall [*3*] supported their specimens on piano wires accurately located at the nodal points. For some reason, perhaps this difficulty in obtaining supports that exercise sufficiently low restraint on a specimen, these methods of test have not been widely used. Kesler and Higuchi [*4*], however, have reported tests in which the logarithmic decrement, in combination with the dynamic modulus of elasticity determined from the transverse resonant frequency of a specimen, was used in predicting the compressive strength of concrete. They report an accuracy of prediction generally within five per cent for the limited tests made.

In more recent reports, Chang and Kesler [*5*,*6*] have reported studies of short-time creep in compression, long-time creep in compression, relaxation in compression, creep in flexure under uniform loads, and creep in flexure under concentrated loads in which reasonably good correlations were obtained between these measured creep values and values predicted on the basis of dynamic modulus of elasticity, logarithmic decrement, and compressive strength of the concrete studied.

Mention should also be made of the possibility of using sustained vibrations for testing concrete in place. Long and Kurtz [7] have reported such tests in which a large auditorium-type loudspeaker was rigidly attached to a concrete wall and driven at a fairly high power level. A pickup was then moved about on the surface of the wall to determine points of maximum vibration. The quantity measured was the velocity of the standing wave, since the frequency of vibration was known and the wave length of the vibration within the concrete could be determined. This and similar techniques have not been widely used, although Pickett [8] has suggested that tests of a similar nature might be useful in testing concrete pavements.

The greatest use of sonic techniques has been made in evaluating the performance of concrete specimens subjected to natural or artificial weathering. They have also been used to study the effect of moisture content and to compare different mixes [3], to investigate the efficiency of various curing compounds [7], and for other purposes [9]. It has been suggested that they may be used to study the setting characteristics of concrete, but such usage has generally proved unsuccessful. With the exception of the work of Kesler and Higuchi [4], most efforts to relate the elastic properties of concrete to its strength have been largely unsuccessful. Somewhat better results have been obtained in efforts to correlate changes in dynamic modulus of elasticity to changes in strength, and in weathering studies it is fairly common to equate a 30 per cent decrease in dynamic modulus to a 50 per cent decrease in flexural strength.

Unfortunately all of the techniques discussed above, with the possible exception of the last, are subject to two major limitations. The methods are basically applicable to specimens of relatively small size and are of little value in studying the behavior of concrete in place. Further, because of the complexity of the calculations involved in computing the constants D, C, and B, specimens must be either cylinders or prisms of uniform cross section (square or rectangular). It is perhaps due largely to these restrictions that recent attention has been directed toward the development of devices for determining pulse velocity in concrete.

PULSE TRANSMISSION TESTS

The application of pulse transmission techniques to the testing of concrete is believed to have had its origin with Obert [10]. Tests were made on concrete replacement pillars in mines and involved the use of two geophones, two-high-gain amplifiers, and a camera with a moving film strip. Two holes, approximately 20 ft apart vertically, were drilled into the pillars. The geophones were placed in the backs of the holes and the holes filled with cotton waste. A hammer blow was struck at the base of the pillar, and at the same time the camera lens was opened and the moving film strip exposed. After the film was developed, the transit time of the impulse in travelling from one geophone to the other was determined by measuring the distance between the two signals on the film, the speed of motion of the film having been carefully controlled. The velocity could then be calculated.

Long and Kurtz [7, p. 1067] reported performing somewhat similar experiments with a Shepard seismograph in which the longitudinal velocity of the pulsation created by a single impact was measured between arbitrarily placed geophones. They stated that only very limited experiments of this nature had been conducted but that the method appeared to hold great promise providing the apparatus could be adapted to the measurement of much shorter time intervals than those of which the seismograph was capable.

Long, Kurtz, and Sandenaw [*11*] undertook further investigations along these lines and in 1945 reported on the instrument and technique that resulted from their work. The apparatus consisted of two vibration pickups (in the form of phonograph cartridges), two amplifiers, two thyratron tube circuits, and a ballistic galvanometer circuit. The impact of a hammer blow was impressed upon the concrete in a horizontal direction, approximately in line with the two pickups. The energy impulse thus generated actuated the first pickup, the voltage from which energized the first thyratron and started a flow of current through the galvanometer. When the energy impulse reached the second pickup, the voltage from its amplifier ionized the second thyratron and cut off the flow of current. The deflection of the galvanometer was directly proportional to the time required for the wave to travel the distance between the two pickups.

In a discussion of this paper, the substitution of an electronic interval timer for the ballistic galvanometer was suggested. This device consists of a capacitor which begins to charge when the first thyratron is ionized and stops charging when the second is ionized and a vacuum-tube voltmeter which measures the charge. The meter may be calibrated directly in units of time, thus eliminating the necessity for computations involving the magnitude of the current flowing through the galvanometer. This device was found to be more reliable than the ballistic galvanometer for field use. An instrument very similar to that suggested is now commercially available in the United States under the name of the Electronic Interval Timer.

Subsequent investigations in this country and abroad have resulted in the development of a number of other devices quite similar in most respects to the Electronic Interval Timer. These include the Micro-timer developed by the U.S. Bureau of Reclamation, the Condenser Chronograph developed by the Danish National Institute of Building Research, and devices developed at the National Physical Laboratory, South African Council for Scientific and Industrial Research, and the Laboratoires du Batiment et des Travaux Publics, France. All make use of either hammer blows or small explosive charges to generate the impulse.

In 1946 the Hydro-Electric Power Commission of Ontario, Canada, in an effort to develop a technique for examining cracks in monolithic concrete structures, began a series of studies which resulted in the construction of an instrument known as the Soniscope. The device consists basically of a pulse generator using piezoelectric crystals, a similar pulse receiver, and electronic circuits which actuate the pulse generator, provide visual presentation of transmitted and received signals on a cathode-ray tube, and accurately measured the time interval between the two. Development of this instrument was first reported to Committee 115, Research, of the American Concrete Inst. in 1948. A more complete report was published by Leslie and Cheesman [*12*] in 1949.

The physical and electrical features of the Soniscope have passed through several stages of improvement since 1947, and a number of these instruments have been built by various laboratories in the United States and Canada. In its latest form the Soniscope is patented and commercially available through a firm in Chicago, Ill. It may also be obtained from a Canadian firm. Considerable use of the instrument has been reported in Canada [*13*] and the United States [*14*].

During approximately the same time that the Soniscope was being developed in Canada and the United States, work of a similar nature was being conducted in England. These investigations resulted in the development of an instrument known as the Ultrasonic Concrete Tester. This instrument and the uses to which it has been put have been described at length by Jones [*15*,*16*]. The Ultrasonic Concrete Tester differs from the Soniscope primarily in the much higher frequency used within the transmitted pulse and in the repetition rate which is about three times as great as that of the Soniscope. These changes improve the accuracy of measurement on very small specimens but limit the usefulness of the instrument for field testing, since the high frequencies suffer much greater attenuation in passing through concrete than do the lower ones. The maximum range of the Ultrasonic Concrete Tester is believed to be about 7 ft, whereas that of the Soniscope in testing reasonably good concrete is 50 ft or more.

Several devices have been reported which incorporate some of the features of the Soniscope and some of the Electronic Interval Timer. In these instruments the impulse is generated by a mechanical blow, frequently that of a spring-loaded hammer operated by a motor-driven cam at the rate of about five blows per second. The receiver may be any one of a number of types of pickup. Transmitted and received signals are displayed on a long persistence cathode-ray oscilloscope containing a calibrated time base for determining the transit time between signals. Development of one such device has been reported in the United States and another in France.

The devices mentioned above and the uses to which they have been put have been described in detail by Whitehurst [*17*] and in several recent publications of the Réunion des Laboratoires d'Essais et de Recherches sur les Matériaux et les Constructions [*18*].

Whereas the use of the sonic tests has been restricted primarily to the evaluation of specimens undergoing natural or artificial weathering and the techniques for such use have been largely standardized, pulse transmission techniques have been applied to concrete for many purposes and, in most areas of investigation, only limited agreement has been reached concerning the significance of test results. The quantity actually measured by all of these instruments is the transmission time of an impulse passing through the concrete under test. If the path length between generator and receiver is known or can be determined, the velocity of the pulse may easily be computed. It is in the interpretation of the meaning of this velocity and in its use for determining various properties of concrete that agreement is incomplete. The technique is as applicable to concrete in place as to laboratory-type specimens, and results appear to be unaffected by the size and shape of the concrete tested, within the limits of transmission of the instrument employed, provided care is taken in testing very small specimens. This, of course, is a highly desirable attribute and, in many respects, makes the pulse transmission techniques more useful than those involved in sonic testing.

Because of the fundamental theoretical relationship between pulse techniques and resonant frequency techniques, there is a strong inclination for users of the pulse technique to endeavor to compute the dynamic modulus of elasticity from the results of the tests. Theoretically, such values of modulus should be the same as those determined by resonant frequency tests upon the same specimens. The experience of several investigators [*19*,*20*], however, has shown that on some occasions this is true and on others it is not. Because of these presently unexplainable differences, most of those experienced in the use of pulse velocity techniques are inclined to leave their results in the form of velocity without attempting to calculate moduli therefrom.

If the modulus of elasticity is to be computed from the pulse velocity, the relationship generally recommended is:

$$E = V^2 \rho \frac{(1 + \mu)(1 - 2\mu)}{(1 - \mu)} \quad (8)$$

where:

E = dynamic modulus of elasticity,
V = longitudinal pulse velocity,
ρ = mass density, and
μ = Poisson's ratio.

This equation relates modulus to pulse velocity and density in an infinite medium and presumably should apply only to mass concrete. The experience of most investigators, however, has been that even for very small laboratory specimens this relationship gives better results than do those applying to either slabs or long slender members. Leslie and Cheesman [*12*] have suggested that best results are obtained if, for concretes having unit weights in excess of approximately 140 lb/ft,^{3} the value of Poisson's ratio is assumed to be 0.24. If this is done, Eq 8 is reduced to:

$$E = 0.000216V^2d(0.848) \quad (9)$$

where d = weight of concrete, lb/ft.^{3}

The factor 0.848 represents the correction for Poisson's ratio, taken in this case to be 0.24.

Fundamentally all of the pulse velocity techniques and equipment developed for use on concrete result in the measurement of the compressional, or longitudinal, pulse velocity. In rare cases, with the Soniscope-type instrument, it is possible to measure the transverse pulse velocity and the Rayleigh pulse velocity as well, when the test is being made across a corner of a structure and the path length involved is fairly long (in the order of 15 to 20 ft). Under these circumstances it is theoretically possible to calculate Poisson's ratio directly from any two of the measured velocities. The occurrence is sufficiently rare with present equipment, however, to merit no more than mention at this time.

The use of pulse velocity techniques for testing concrete has been suggested for evaluating the strength of concrete, its uniformity, its setting characteristics, its modulus of elasticity, and the presence or absence of cracks within the concrete. There appears to be little question of the suitability of such techniques to determine the presence, and to some extent the magnitude, of cracks in concrete, although it has been suggested that if the cracks are fully water-filled their locations may be more difficult to ascertain. In all of the other fields of investigation, independent investigators have reported widely different degrees of success through the use of these techniques [*17*].

The most complete report of the experiences of users of pulse velocity techniques in the United States and Canada may be found in "Effects of Concrete Characteristics on the Pulse Velocity—A Symposium," *Bulletin No. 206*, Highway Research Board, 1958.

Experiences in the use of pulse velocity measurements for evaluating concrete quality have been reported in many other countries, notably Great Britain and Russia. In some instances investigators in these countries have appeared to have greater confidence in the use of such techniques for acceptance testing than has been the case in the United States.

Jones [*16*] has reported values of minimum velocity considered acceptable for concrete for certain specific structural purposes in Great Britain as follows:

Prestressed concrete—T sections	15,000 ft/sec
Prestressed concrete—anchor units	14,300 ft/sec
Reinforced concrete frame building	13,500 ft/sec
Suspended floor slab	15,500 ft/sec

In a later publication [*21*], however, he stated that "pulse-velocity has rarely been used as an acceptance criterion for structural concrete because it is usually too difficult in site-work to ensure that the control of mix proportions, size and type of coarse aggregate remains uniform."

Several reports are available of the use of pulse velocity techniques in Russia. Filina [*22*] has reported on some 80 various items of equipment for testing concrete which are in use in the USSR, including those making use of ultrasonics and vibrations.

Zashchuk and Nefedova [*23*] have described the design and operation of ultrasonic testing equipment used in connection with the construction of the Moscow Ring Road. They state that toward the completion of the road ultrasonic methods replaced all other techniques.

It is generally agreed that very high velocities are indicative of very good concrete and that very low velocities are indicative of poor concrete. It is further agreed that periodic, systematic changes in velocity are indicative of similar changes in the quality of the concrete. Beyond these areas of agreement, however, it appears that the investigator must have a rather intimate knowledge of the concrete involved before attempting to interpret velocities as measures of strength or other properties of the concrete. This is particularly true if the aggregate involved is a lightweight aggregate.

Aside from the applications of dynamic testing described above, several other possible applications of the described techniques are currently under investigation. Among these are the testing of driven piles—to determine whether they have been damaged in driving—and the measurement of pavement thickness. In the latter matter, some success has been reported by Muenow [*24*]. The technique developed involves measurement of resonant frequency in the thickness dimension of the slab and pulse velocity in the slab. From these two measurements slab thickness may be calculated. Accuracy in the order of five per cent, as compared to the lengths of cores removed from the slab, is claimed.

It is certain that further explorations in the areas outlined above and in others will be forthcoming.

SURFACE IMPACT MEASUREMENTS

Within recent years some measure of success has been achieved in making quantitative measurements that are indicative chiefly of the resistance of concrete to the rapidly applied blow of a steel hammer or ball. Hence, these measurements may provide another means of surveying concrete in structures to determine its uniformity as affected by composition, placing, curing, or exposure. Although there is a pronounced tendency on the part of users of these devices to express the observations in terms of estimated strengths, it may be found that the results are expressed more significantly as differences either with respect to various locations in a structure or a series of production units or in designating changes resulting from hydration or exposure. Such measurements may be considered conveniently under the headings of rebound and indentation tests.

For several years use has been made of spring-actuated hammers whose rebound movement after striking the surface of concrete can be recorded [*25,26*]. These Schmidt test hammers are of two types. Model I has external springs, whereas in Model II the spring mechanism is enclosed within the barrel of the instrument. In Model I the hammer is cocked against the spring and released by a trigger. In Model II the hammer is pushed against the surface of the concrete to compress the spring by a predetermined amount. At this point the spring is released automatically, thereby driving a ram against the hammer.

Among the earlier uses of impact indentation tests was one described in the *Concrete Manual* of the U.S. Bureau of Reclamation [*27*]. It was felt that strengths in the 500 to 2000 psi range could be estimated with sufficient accuracy to provide a check on the time at which tunnel forms might be removed safely. It was indicated, however, that the test was not so reliable for strengths at later ages or higher levels.

More recently the results of similar indentation tests in Germany have been reported by Gaede [*28*]. The instruments used were a pendulum hammer, which was restricted to vertical surfaces, and the Frank spring hammer, generally similar in its operation to the Model II Schmidt hammer. Both hammers may be caused to deliver either half of full blows. In addition, it is indicated that a half-size indentor should be used when the regular one makes insufficient impression in high-grade concrete.

Several additional impact tests have been discussed by Voellmy [*29*]. These are not sufficiently well known on this continent, however, to permit their evaluation at this time.

In view of the important influence of surface and near-surface conditions on impact tests, the instructions supplied with these devices deal in some detail with the preparation of surfaces for testing, the minimum number of blows per test, and the recommended procedure for analyzing the results. Most investigators have found that small test specimens have insufficient mass to provide an adequate reaction. Accordingly, they have reported clamping specimens in a testing machine under a load of a few thousand pounds in order to provide satisfactory restraint. Others have reached the conclusion that finished, horizontal surfaces do not represent the underlying concrete as closely as do formed, vertical surfaces. The importance of this difference will depend, of course, upon whether one is interested chiefly in the condition of the surface or that of the concrete as a whole. It is obviously necessary to keep these and other details of procedure constantly in mind in order to secure consistent and usable results.

Originators of the spring-driven rebound and indentation hammers have established general curves based on fairly extensive tests and a wide range of concretes. These calibration curves provide some guidance for users of the hammers, but it has been found that a correlation curve for the specific concrete is much more useful and trustworthy for control of a given job. This is obviously the case, since it evaluates the influence of aggregate type and size distribution.

Numerous investigators have developed strength correlation data showing a considerable range in coefficients of variation depending largely upon their control of variables in the concrete and on the details of their testing techniques. Experiences, reported largely through personal correspondence, of those calibrating and using various rebound and indentation hammers indicate that, with careful calibration and test control, values of compressive strength predicted from weighted mean rebound numbers or indentation diameters will generally agree with measured compressive strengths within 15 per cent, with the majority agreeing within 10 per cent. Some investigators have claimed accuracy of prediction within considerably narrower limits. It has been reported that the degree of correlation decreases as strength or age of the concrete increases, and it has been further suggested that variations in moisture content of the concrete may have a significant effect upon rebound or indentation test results.

On the basis of recent work at the U.S. Bureau of Reclamation, Mitchell and Hoagland [*30*] recommended that special calibrations be provided for each mix or change of aggregate, when strength was to be estimated on the basis of impact hammer tests, and that hammer tests on weak or young concrete be minimized because of the possibility of producing significant surface blemishes.

Conclusions of investigators in other countries appear to generally parallel those published in the United States. Victor [*31*] has recently stated that it is possible to develop a calibration curve when the average values of large numbers of results are plotted, but that the scatter of results is too wide to facilitate any reliable inference from any individual test result.

SUMMARY

It has been the experience of users of dynamic testing that many factors influence the results in varying degrees. Recognizing these influences and making proper allowances for them are basic requirements in evaluating such test data.

Usually the most pronounced influence on results is the type of aggregate used in the concrete because of the wide ranges possible in elasticity and density. Frequently the maximum size of coarse aggregate must be considered in comparing different series of tests. Other factors influencing results include moisture content, temperature, density, reinforcement, type of cement, aggregate-cement and water-cement ratios, and admixtures. The most exhaustive investigation of these factors reported to date is that of Jones [*16*].

It appears that the sustained frequency tests have their main application in tracing the course of deterioration in specimens subject to weathering or exposure tests. In general, application of these techniques to structures, including pavements, is hampered by boundary effects, power requirements, and difficulties in the interpretation of measurements on any but the most elementary forms.

The major applications of pulse velocity tests on concrete are to establish the degree of uniformity, or lack thereof, throughout a structure, to follow progressive changes in the quality of concrete in either specimens or structures, and to determine the presence or absence of cracking in monolithic concrete. Velocity tests on concrete are not hampered significantly by size and shape effects. The level of effective pulse transmission is the limiting condition that will govern the operating range, depending on the characteristics of instrumentation and on the inherent property of concrete to attenuate the impulse. This maximum range may vary from perhaps a few inches in unset concrete to 50 to 60 ft in sound concrete of high quality. The attenuation of the impulse may lead to important errors, particularly in the case of those devices which do not provide for visual examination of transmitted and received signals.

With respect to the prediction of values for other properties of concrete on the basis of the results of dynamic tests, it is both desirable and proper to compute the dynamic modulus of elasticity or rigidity from the appropriate resonant frequency. Such computation is necessary if results of tests on specimens of different sizes and shapes are to be compared. Since both the resonant frequency and the weight of the specimen can be measured directly and Poisson's ratio does not enter heavily into the computations (if longitudinal resonance is measured), it is believed that there is little danger of the introduction of significant error in making these computations. The use of resonant frequency techniques for predicting other properties of concrete does not appear to be well supported by data presently available, although the recent work of Kesler [*4*] and Chang and Kesler [*5,6*] indicates that two dynamic parameters may define the strength of concrete and its creep characteristics.

With respect to pulse velocity techniques, there seems to be very little reason for computing anything other than pulse velocity from the results of such tests. The computation of dynamic modulus of elasticity from pulse velocity requires a knowledge of both the unit weight of the concrete and Poisson's ratio, both of which values at least in the case of tests on structures, must be estimated. Since results of these tests are not dependent upon size and shapes of the concrete tested, direct comparisons may be made between tests made on different concretes or different sections of the same concrete. Batchelder and Lewis [*20*] have shown that the velocity itself correlates better with the results of resonant frequency tests on laboratory specimens than does the modulus of elasticity computed from velocity tests. No evidence has yet been presented to suggest that any better relationship exists between pulse velocities and other properties of the concrete than between resonant frequencies and these properties.

It is believed that hammer impact tests which can be applied directly to concrete in place and which have the desirable attributes of ease of application and low equipment cost can provide a useful means of quality control and inspection in many instances. It must be borne in mind that the measurements reflect conditions at or near the surface of a structure or member and hence may or may not be indicative of the underlying concrete.

With the present state of knowledge of the variations inherent in these testing techniques and the recognition of other factors influencing the results, it appears essential in attempting to estimate the absolute level of strength of a particular concrete to have calibration data for that type of concrete. However, in spite of these limitations, with careful adherence to recommended test procedures and with discreet and judicious interpretation of test data, the impact tests are potentially capable of providing much useful guidance for the users.

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Hardened Concrete

VOLUME CHANGES

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Concrete is an important and widely-used constructional material because of its excellent combination of desirable properties, but it is subject to appreciable volume changes which may, under certain circumstances, result in poor performance, cause rupture, or aid the action of destructive agents through the development and growth of cracks. Volume changes in concrete due to variations of temperature, humidity, and stress are partly or entirely reversible, but volume changes due to destructive chemical and mechanical action are not reversible and are cumulative as long as the action continues.

Unrestrained volume changes in concrete due to variations in temperature, moisture, and stress are generally of small concern. When volume changes are restrained by foundations, connecting members, or reinforcement, stresses are produced in the concrete which may cause distress and even failure. Since concrete is weaker in tension than in compression, restrained contraction are usually more important.

While in a general way the causes of volume changes and the reactions of concrete to these causes are known, it is still not possible to build structures such as bridges, buildings, dams, and pavements with assurance that they will not crack. However, if proper attention is given to all of the many variables that influence the behavior of concrete, it is possible to build these concrete structures so that they are relatively crack free and satisfactorily resist the action of destructive agents.

The magnitude of volume changes is usually given in linear rather than volumetric units because linear changes can be measured easily and are of primary interest to engineers. A given length change may be reported, for example, as 800 millionths of an inch per inch, or 0.08 per cent, or 0.96 in./100 ft.

Test Methods for Determining Volume Changes

It is impossible to standardize all volume change tests made on concrete because of the wide variety of conditions encountered in research. Simple routine tests, such as those for the determination of unsoundness caused by the hydration of uncombined lime and magnesia and those for the determination of the possible reaction between aggregates and cements having a high alkali content, will be considered later. In addition, ASTM Test for Length Change of Cement Mortar and Concrete (C 157 – 64 T) provides a method for routine determination of the volume change of unstressed cement mortar and concrete specimens at room temperature. The test consists of casting mortar prisms, 1 in. square and 11 in. long, or concrete

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prisms, 3 in. square and 11 in. long, containing aggregate up to 1 in., in a horizontal position. The specimens contain a stainless steel gage plug at the center of each end which projects $\frac{1}{8}$ in. beyond the end. The distance between the innermost ends of the gage plugs, the gage length, is 10 ± 0.10 in. Specimens are kept in the molds for $23\frac{1}{2} \pm \frac{1}{2}$ hr, or longer if necessary, to prevent damage, removed from the molds, and moist cured for 28 days. After curing specimens may be stored in water at 73.4 ± 3 F, or in air at 73.4 ± 2 F and a relative humidity of 50 ± 4 per cent with air movement controlled to result in a uniform rate of evaporation of water from an atmometer or beaker, to determine linear expansion due to moist storage or linear contraction due to air storage. Length changes are obtained with a dial micrometer.

Unless information on the volume change of concrete during the setting period is wanted, the first length reading is usually taken after 24 hr, and readings are taken at additional ages until the desired information has been obtained. Length readings are usually taken over gage lengths varying between 1 and 20 in. with dial comparators, mechanical strain gages, optical comparators, and electric resistance gages. Length readings may be taken over a wide range of temperature and under variable storage conditions such as moist, sealed, dry with different relative humidities, and various cycles of dry and moist.

ASTM Test for Volume Change of Concrete Products (C 341 – 54 T) provides a routine standardized procedure for determining the volume changes of concrete products such as masonry units, concrete pipe, cast stone, or sawed sections representing these products when stored in air or water under various conditions. ASTM Test for Drying Shrinkage of Concrete Block (C 426 – 61 T) is intended to provide a routine standardized procedure for determining the drying shrinkage of concrete block, brick, or other concrete products. Drying shrinkage in this method is defined as the change in linear dimension due to drying from a saturated condition to a constant weight and length under specified conditions (in air at a constant, uniform temperature of 122 ± 2 F, and a relative humidity of 17 ± 2 per cent).

VOLUME CHANGES IN FRESH CONCRETE

Volume changes in freshly mixed concrete are due to water absorption, sedimentation (bleeding), cement hydration, and thermal change, and are influenced by the temperature and humidity of the surrounding atmosphere. Absorption of water by the aggregates and reaction between the water and the cement both act to decrease the volume. In bleeding, the solid portions of the mix settle while the clear water rises to the top. This action starts very shortly after the concrete has been placed and continues until maximum compaction of the solids, particle interference, or setting brings it to a close. Profuse bleeding, together with rapid evaporation or leaky or absorbent forms, will result in excessive setting shrinkage which, in extreme cases, may be as high as 1 per cent by volume. Setting shrinkage may be minimized by the use of saturated aggregates, low cement content mixtures, moist and cool casting conditions, tight and nonabsorbent forms, and shallow lifts in placing.

Plastic shrinkage and plastic shrinkage cracking occur in the surface of fresh concrete within the first few hours after placement. The principal cause of this type of shrinkage is an excessively rapid evaporation of water from the concrete surface, and it occurs most frequently in slab and pavement construction exposed to the hot sun and drying winds. Plastic shrinkage cracks may have considerable depth and are usually straight line cracks without any definite symmetry or pattern, but they may have a crow's-foot pattern. Corrective measures to prevent plastic shrinkage cracking are all directed toward reducing the rate of evaporation or the total time during which evaporation can take place.

VOLUME CHANGES IN HARDENED CONCRETE

Undesirable Chemical and Mechanical Attack:

Concrete is subjected to many types of chemical and mechanical attack that act to shorten its useful life. While the mechanism of destruction varies and may be quite complicated, signs of its action are generally first evident as expansions, and as the action continues the expansions increase until disintegration occurs. Some of the more common destructive agents or actions to which concrete is subjected include sewage of high acid or sulfide content, sulfate waters, electrolysis, sea water, fire, freezing and thawing, expansion due to hydration of uncombined lime or magnesia or both, expansion due to a reaction between cements having a high alkali content and certain siliceous aggregates, expansion due to reactions between cement and certain sand-gravel aggregates, and expansion due to a reaction between cements having a high alkali content and certain argillaceous dolomitic limestones. Difficulties in service may also be due to dimensional instability of the aggregates and to a loss in bond between aggregates and cement paste. A complete discussion of each of these actions is outside of the scope of this paper, but additional consideration will be given to the most important.

Excessive expansion of concrete due to the hydration of the uncombined lime and magnesia present in cement is called unsoundness. The autoclave test provides quantitative information on the expansion due to the hydration of free calcium oxide and magnesium oxide. The test as given in ASTM Test for Autoclave Expansion of Portland Cement (C 151) consists in subjecting 1 by 1 by $11\frac{1}{4}$-in. neat cement bars of normal consistency, moist cured for 24 hr ± 30 min, to the action of steam under a pressure of 295 ± 10 psi for a period of 3 hr. The steam pressure must be raised to the required value in 45 to 75 min, and after the 3 hr steaming period the pressure should be released to a value less than 10 psi within $1\frac{1}{2}$ hr. Linear expansions are measured by a dial gage or a micrometer comparator over an effective gage length of 10 in. A maximum expansion of 0.80 per cent is allowed in present ASTM standards for cement Types I, II, III, IV, V, IA, IIA, and IIIA, and a maximum value of 0.20 per cent expansion is allowed for Types IS and ISA. The autoclave expansion for natural cement Types N and NA is obtained by determining the autoclave expansion of a blend of 25 per cent natural cement and 75 per cent portland cement (Type II unless otherwise specified by the purchaser). The maximum value is 0.80 per cent. Because of large expansions obtained in many structures made with masonry cements containing high percentages of unhydrated magnesium oxide, it is required that the autoclave test be used for masonry cement and that the maximum allowable expansion be 1.0 per cent.

Reactions between certain highly siliceous constituents of aggregates and cements having high alkali contents result in strength loss, excessive expansion and cracking, and disintegration. One method of determining the potential alkali reactivity of cement-aggregate combinations is given in ASTM Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227). The method consists of making 1 by 1-in. prisms, with an effective gage length of 10 in., of 1 part of the cement to be used to 2.5 parts of graded aggregate, by weight, with enough water to produce a flow of 105 to 120 (as determined in accordance with ASTM Test for Compressive Strength of Hydraulic Cement Mortars (C 109)). The prisms are stored for 24 ± 2 hr in a moist room at 73.4 ± 3 F, measured for length in a comparator, placed on end, over but not in contact with water in sealed containers maintained at 100 ± 3 F, and again measured for length after cooling to 73.4 ± 3 F. The expansion for any period of time may thus be determined. A chemical method for determining the potential reactivity of aggregates when used with high alkali cements is given in ASTM Test for Potential Reactivity of Aggregates (Chemical Method) (C 289), which is based on the amount of reaction of the aggregate with a 1N sodium hydroxide solution under controlled test conditions. Alkali-aggregate reactions may be controlled or reduced by: using nonreactive aggregates, using cements with an alkali content less than 0.60 per cent expressed as sodium oxide (Na_2O) equivalent, using certain finely ground pozzolanic materials which react chemically with the alkalis before they attack the reactive aggregates, providing exposure conditions that cause the concrete to become and remain relatively dry, and using other admixtures. Outstanding reductions in expansions of laboratory specimens due to alkali-aggregate reaction have been obtained with lithium and barium salts [*1*].[2] Air entraining agents also act to decrease expansion due to alkali-aggregate reaction, but they appear to vary in effectiveness and become less effective as the severity of the expansive reaction increases [*2*].[3]

An alkali-carbonate reaction, similar in certain respects but different in others to the alkali-silica reaction, is the reaction between some argillaceous dolomitic limestones and high-alkali cements [*3,4*]. Under moist conditions this reaction causes excessive expansion and cracking of concrete. Standard ASTM tests for alkali-aggregate reaction do not always detect this type of reaction, but it may be detected by another test which also detects alkali-aggregate reaction. This test consists of exposing concrete prisms made with the questionable aggregate and a high alkali cement to an atmosphere at 73 F and 100 per cent relative humidity and noting the amount of expansion [*3*]. A commonly used test to detect alkali-carbonate reaction consists of storing aggregage prisms ($1\frac{1}{2}$ by $1\frac{1}{2}$ by $5\frac{3}{4}$ in.) in a 2 *M* alkali hydroxide (equal molar parts of NaOH and KOH) solution. Expansions of the questionable aggregate prisms are then compared with those obtained on companion prisms of known sound limestone [*5*]. While firm criteria are not yet established it appears that aggregate having linear expansions in excess of 0.03 to 0.04 per cent greater than the comparison aggregate at ages up to ten months should be regarded with concern. Apparently this undesirable reaction may be partially controlled when the reactive aggregate must be used by using a cement with an alkali content lower than 0.40 per cent, expressed as Na_2O equivalent, and by keeping the concrete relatively dry. The usual alkali-silica reaction inhibitors such as pozzolans and lithium chloride are not effective in controlling the alkali-carbonate reaction.

Concrete made with sand-gravel aggregates from Kansas, Nebraska, and

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

[3] See also p. 487.

portions of several adjacent states may be subjected to deleterious expansion and deterioration under certain conditions. It has been suggested that this process involves physical and chemical phenomena that have not yet been defined [*6*]. Apparently the alkali-aggregate reaction is involved to a varying degree, and other factors, such as delayed hydration of free magnesia and rupture of the cement-aggregate bond, may also be present. Replacement of 30 per cent or more, by weight, of the questionable aggregate with crushed limestone is used for partial control of the undesirable expansion and cracking [7].

Large dimensional changes of concrete have also been associated with aggregates that undergo excessive drying shrinkage in air and large expansion in water [*8*]. Certain sandstones usually containing clay minerals have caused this type of difficulty in the Union of South Africa, and certain basic igneous rocks of the basalt and dolerite type have caused distress in Scotland. Apparently in the past, tests other than those concerned with dimensional stability have eliminated largely the use of inferior aggregates of this type in the United States. The cause of the dimensional instability of the aggregate itself under conditions of wetting and drying is uncertain. The presence of clay minerals with expansive lattices such as montmorillonite offers only a partial explanation of the dimensional instability since large dimensional changes have also been observed in an aggregate that does not contain such clay minerals. Further, it is believed that the shrinkage and expansion of the mortar is not related simply to the dimensional change of the rock and that complex phenomena are involved.

Autogenous Action:

Autogenous volume changes of concrete are the result of cement hydration and do not include changes due to variations in total moisture, temperature, and load. Autogenous volume changes may cause expansions or contractions, depending on the relative importance of two opposing factors: (1) expansion of new gel due to the absorption of free pore water, and (2) shrinkage of the gel due to extraction of water by reaction with remaining unhydrated cement. In most instances the initial expansions obtained during the first few months do not exceed 0.003 per cent, while the ultimate contractions obtained after several years usually do not exceed 0.010 per cent. These volume changes are especially important in the interior of mass concrete where little or no change of the total moisture content is possible [*9*].

Results of tests indicate that autogenous volume changes are influenced by the composition and fineness of the cement, the amount of mixing water, mixture proportions, curing conditions, and time. It appears that the magnitude of the autogenous volume change increases as the fineness of the cement and the amount of cement for a given consistency are increased. Ultimate contractions appear to be somewhat greater for low heat cements (Type IV) than for normal portland cement (Type I).

Thermal Changes:

Unrestrained concrete expands as the temperature rises and contracts as it falls. An average value of the coefficient of thermal expansion normally used is 5.5 millionths per deg F, which fortunately is close to the value for steel. Consequently reinforced-concrete structures function satisfactorily even over fairly large temperature variations. While the coefficient of thermal expansion frequently is close to the average value, it may vary between 2.5 and 8.0

millionths per deg F, depending to some extent on the richness of the mix, the type of aggregate, and the moisture content of the concrete, but primarily on the thermal coefficient of the aggregate used [*10*].

The coefficient of thermal expansion of hardened cement paste varies between 5.0 and 12.5 millionths per deg F and increases about 25 per cent as the fineness is increased from 1200 to 2700 cm^2/g (Wagner). Oven-dried and vacuum-saturated specimens have similar coefficients, but the apparent value for specimens with an intermediate moisture content may be almost twice as large.

Thermal expansion of concrete is influenced greatly by the type of aggregate used because of the large differences in the thermal properties of the various types of aggregates and even for aggregates of a given type but from different sources. Siliceous aggregates, such as chert, quartzite, sandstone, and some highly impure limestones, have thermal coefficients of expansion between 4.5 and 6.5 millionths per deg F, while the coefficient for purer limestones, basalt, granite, and gneiss have values between 1.2 to 4.5 millionths per deg F. Single crystals of minerals such as quartz, feldspar, and calcite, or rocks composed of such minerals with the crystals in parallel orientation have coefficients along the various axes that may be quite different. Feldspar, for example, has values of 9.7, 0.5, and 1.1 millionths per deg F along three different axes. A fair value of the coefficient of thermal expansion for a concrete made with specific materials may be computed by using weighted averages of the coefficients of the aggregate and the hardened cement paste.

The differences in the thermal coefficients of the constituent materials in concrete may cause a thermal incompatibility between the coarse aggregate and the cement mortar. It is agreed generally that this thermal incompatibility has some effect on durability, but its relative importance in comparison with those caused by other factors is in some doubt.

Thermal changes are important in mass concrete where cracking is generally due to cooling of the concrete from the maximum temperature reached to the stable temperature. Drying shrinkage in mass concrete is important only at the surface. The maximum concrete temperature is dependent on the initial concrete temperature, the heat of hydration of the cement, the outside temperature, the rate of construction, and the specific heat and thermal conductivity of the concrete. Thermal changes in mass concrete are kept as low as possible by the use of low-heat portland cements and also by artificial refrigeration.

Thermal changes are also important in pavements since differential gradients at night frequently cause a shortening of the top surface relative to the bottom surface, thus tending to lift the slab ends above the subgrade with the result that its ability to support traffic loads without cracking is impaired [*11*].

Continuous Moist Storage:

Moist-cured concrete begins to expand after the setting shrinkage has taken place because of progressive hydration of the cement and the gel formation between cement particles. The expansion of concrete due to continuous moist storage is relatively low for the various types of cement, although it is slightly higher for Type I than for Types II, III, IV, and V [*8*]. Mortars and concretes containing pozzolanic material as cement replacements usually expand slightly more under continuous moist storage than do comparable mortars and concretes made without pozzolanic replacements. Additions of gypsum above the optimum value also increase expansions under moist storage. The amount of cement in a given mixture is of much greater importance than the type of cement, since the expansion of a neat paste is about twice that of an average mortar and the expansion of the mortar is about twice that of an average concrete.

The rate of expansion decreases with the period of moist storage and becomes very small after several years. The ultimate amount of expansion is usually less than 0.025 per cent. For comparative purposes it may be noted that the expansions accompanying immersion over a period of several years are about one third of the contractions of the same concrete air dried for the same period.

The volume change of concrete protected against loss or gain of water is small and is usually less than 0.005 per cent expansion or contraction, depending on the type of cement, temperature, and other conditions.

Drying Shrinkage:

Drying shrinkage takes place concurrently with carbonation shrinkage (due to the reaction between carbon dioxide and the constituents of cement). Most reported data do not distinguish between the two effects, and the reported shrinkage is usually designated as drying shrinkage. Carbonation shrinkage may be very small, as in the case of structural concrete made with sand and gravel aggregates kept in a continually wet or dry condition, or it may approach the magnitude of drying shrinkage with relatively porous concrete dried to equilibrium in air at 50 per cent relative humidity.

Drying shrinkage of concrete is caused principally by the contraction of the calcium silicate gel in the hardened cement-water paste when the moisture content of the gel is decreased. Among the more important factors that influence drying shrinkage are the unit water content of the concrete, the composition of the cement, the quality and quantity of paste in the concrete, the characteristics and amounts of admixtures used, the proportions of the mixture, the mineral composition of the aggregate, the maximum size of the aggregate, the size and shape of the concrete mass, the amount and distribution of reinforcing steel, the curing conditions, the length of the drying period (carbonation effects are present under relatively long drying periods), and the humidity of the surrounding air [*13*].

In addition, usual methods of measuring drying shrinkage may be misleading if internal cracking of the concrete has occurred, because internal cracking allows shrinkage to occur without fully reducing the over-all length. Under such conditions reported drying shrinkages may be too low. However, internal cracking which is not evaluated by the standard tests for drying shrinkage may be important in the serviceability of concrete under actual conditions of use.

The most important single factor affecting shrinkage is the amount of water per unit volume of concrete. Consequently, concrete with a wetter consistency will shrink more than one with a dry or stiff consistency because the wetter consistency is obtained by the use of a higher water-cement ratio, by a greater quantity of paste, or a combination of the two. The quantity of cement per unit volume of concrete appears to have an erratic effect on shrinkage, since the effect of the larger quantity of paste in a rich mix is offset by the higher water-cement ratio of the paste in a lean mix. Generally, for given materials and a constant unit water content, the cement content has only a minor effect on drying shrinkage. However, for relatively rigid aggregates such as dolomite and granite there is a tendency for drying shrinkage to increase as the cement content increases [13].

The importance of the quality and the quantity of cement paste in the drying shrinkage of concrete is evident from the fact that cement paste in concrete, if not restrained by aggregate, shrinks from 5 to 15 times as much as the concrete. The quality of the paste is primarily a function of the water-cement ratio and the composition and fineness of the cement. A paste with a water-cement ratio of 0.56, by weight, shrinks about 50 per cent more than one with a water-cement ratio of 0.40. The effect of the fineness of the cement on drying shrinkage does not appear to be indicated as clearly, but in general finer cements exhibit slightly greater shrinkage values. In considering composition effects it appears that a given amount of tricalcium aluminate contributes most and tricalcium silicate least to shrinkage and that dicalcium silicate has an intermediate effect. Gypsum has a major effect on shrinkage and for a given cement there is an optimum amount that produces the smallest shrinkage.

The effect of admixtures on drying shrinkage appears to vary for the different types. Conflicting evidence in the literature suggests that when drying shrinkage is important admixtures should be used with care, preferably after evaluation under job conditions. Admixtures that increase the unit water content of concrete may be expected to increase drying shrinkage, but many admixtures that reduce the unit water content do not reduce drying shrinkage. In general, concretes that have some of the cement replaced by pozzolanic materials such as pumicite or raw diatomaceous earth shrink more under continuously dry conditions than concretes without pozzolanic substitutions. Replacement of cement in concrete with up to 30 per cent of low-carbon, high-fineness fly ash results in about the same or slightly lower shrinkage than obtained in concrete made with normal cement [14]. Additions of calcium chloride up to 2 per cent by weight of the cement usually increase drying shrinkage by 10 to 50 per cent. Triethanolamine, frequently used to increase the rate of strength development, increases drying shrinkage [15]. The air entrained, within normal limits, by air-entraining admixtures has little effect on drying shrinkage.

The grading, composition, and the physical and mechanical properties of the aggregate have an important effect on concrete shrinkage, because aggregate particles embedded in cement paste restrain drying shrinkage. Well-graded aggregates with a large maximum size have a low void space and, consequently, require a relatively small amount of paste. Larger maximum sizes of aggregates are effective in reducing shrinkage because they allow lower water contents and because they encourage cracking between particles. Concrete of the same cement content and slump containing $\frac{3}{8}$-in.-max size aggregate usually develops from 10 to 20 per cent greater drying shrinkage than concrete containing $\frac{3}{4}$-in.-max size aggregate, and from 20 to 35 per cent greater drying shrinkage than concrete containing $1\frac{1}{2}$-in.-max size aggregate. The actual amounts are dependent on variables such as aggregate type, length of air-drying period, cement content, and test procedure details [13,16]. The effect of aggregate size may also be noted from the average one-year drying shrinkage values for neat cement, mortar, and concrete of 0.25 to 0.30, 0.06 to 0.12, and 0.03 to 0.08 per cent, respectively. Aside from the question of size, aggregates having a high modulus of elasticity and those having rough surfaces offer greater restraint to shrinkage. Mineral composition is important because, under comparable conditions, concrete made with sandstone, slate, hornblende, and pyroxene may shrink up to two times as much as concretes made with granite, quartz, feldspar, dolomite, and limestone. Concrete made with dirty sand or with unwashed coarse aggregates containing detrimental clay shrinks significantly more than concrete made with clean aggregates.

The size and shape of the concrete mass have a considerable effect on the rate and total amount of shrinkage. In large concrete members, differential volume changes occur with the largest shrinkages found at and near the surface. Because of the large moisture variations from center to surface, tensile stresses are set up at and near the surface, while compressive stresses are developed in the interior. Hence, if the tensile stress near the surface is very high, surface cracks may appear. However, the action of creep may prevent cracking and may cause permanent elongation of the fibers in tension and shortening of the fibers in compression. The rate and ultimate shrinkage of a large mass of concrete are smaller than the values for small size concrete specimens, although the action continues over a longer period for the large mass. Under given drying conditions a 6-in.-thick concrete member might reach equilibrium in one year, while a 12-in.-thick member might require at least four years; the ultimate shrinkage of the larger member, however, might not exceed two thirds of that obtained for the smaller one.

An increase in the length of the preliminary curing period has produced conflicting shrinkage results for neat cement paste specimens, apparently due to variable effects of carbonation during the drying period. It has been shown [*17*] that when the cement paste is protected from carbon dioxide, drying shrinkage increases with the duration of the moist storage period prior to drying. An increase in the length of the preliminary curing period usually has little effect on drying shrinkage of concrete. Prolonged curing increases the modulus of elasticity and reduces the rate of creep at a given stress and, thus, tends to cause cracking under conditions of severe restraint, but the increase in tensile strength that occurs during the same period tends to increase the resistance to cracking. If cracking does take place shrinkage may be diminished. High-pressure steam curing at about 350 F is used effectively in block and precast products plants to greatly reduce subsequent drying shrinkage.

The length of the drying period and the humidity of the surrounding air have an important effect on drying shrinkage. Small specimens of neat-cement paste have shrinkages that increase for at least 20 years and are proportional to the logarithm of the age. The shrinkage of mortars and concretes is usually small *after* 3 years of drying. The rate and magnitude of drying shrinkage increase as the relative humidity of the surrounding air decreases, and the rate increases as air movement past the member increases. Further, pastes, mortars, and concretes that have reached equilibrium under a given drying condition will shrink more if the relative humidity is then decreased or will expand if it is increased.

Drying shrinkage of lightweight concrete can be relatively high, but drying shrinkage in the range observed in normal weight concrete is obtained with careful selection of materials and attention to proportioning of the concrete. Drying shrinkage values of structural lightweight concrete may normally vary between 0.04 and 0.15 per cent and are likely to be more pronounced with concretes containing aggregates that have high rates of absorption and that require high-cement contents for strength. Moist-cured cellular products made with neat cement weighing between 10 and 20 lb/ft^3 may have drying shrinkage values between 0.30 and 0.60 per cent. Autoclaved cellular products that contain fine siliceous material may weigh about 40 lb/ft^3 and have drying shrinkages in the range 0.02 to 0.10 per cent.

Shrinkage of reinforced unrestrained structures produces tension in the concrete and compression in the steel. Increasing the amount of the reinforcement materially reduces the contraction but increases the tensile stress in the concrete. If sufficient reinforcement is used the restraint will be great enough to cause cracking of the concrete. Reinforced concrete structures having normal amounts of reinforcement may have drying shrinkages in the range of 0.02 to 0.03 per cent. Structural concrete slabs reinforced only in tension tend to warp, since the concrete near the top compressive surface shrinks more because of the accumulation of water near that surface during placement, and also because the steel on the tensile side acts to resist shrinkage there.

The ability of concrete to resist cracking is dependent on: (1) the degree of restraint; (2) the magnitude of the shrinkage due to carbonation, drying, and thermal effects; (3) the stress produced in the concrete; (4) the amount of stress relief due to creep; and (5) the tensile strength of the concrete. In order to have good resistance to cracking concrete should have values of drying shrinkage, carbonation shrinkage, thermal coefficient of expansion, and sustained modulus of elasticity as low as possible and a tensile strength as high as possible.

Combined Effect of Unfavorable Factors on Drying Shrinkage:

It has been observed [*17*] and experimentally shown [*15*] that the cumulative effect of the individual factors that increase drying shrinkage can be very large, and that the combined effect is the product rather than the sum of the individual effects. Calculations [*15*] have shown that the use of less favorable construction practices—concrete discharge temperature of 80 F rather than 60 F, a 6 to 7-in. rather than a 3 to 4-in. slump, a $\frac{3}{4}$-in.-max aggregate size rather than $1\frac{1}{2}$ in., and too long a mixing and waiting period—could be expected to increase shrinkage 64 per cent. If, in addition, a cement with high-shrinkage characteristics, dirty aggregates of poor inherent shrinkage quality, and admixtures that increase shrinkage are used the resultant shrinkage could be about five times as large as the shrinkage that would be obtained with the best choice of variables.

Alternate Wetting and Drying:

The effect of the first few cycles of alternate wetting and drying at room temperature on the volume changes of concrete is dependent on the details of the test cycle, but after a few cycles the shrinkage becomes completely reversible although the original length before the first drying is never reached [*18*]. Wetting and drying cycles, combined with alternations of high and low temperatures, cause residual expansions that increase as the number of cycles is increased. One hundred and twenty cycles consisting of 9 hr of oven drying at 180 F followed by 48 hr immersion in water at 70 F and then by 15 hr of air storage at 70 F caused expansions of 0.10 to 0.25 per cent for concrete mixes made with different cements, water-cement ratios, and methods of placement [*19*].

Carbonation Shrinkage:

When concrete is exposed to air containing carbon dioxide it undergoes irreversible carbonation shrinkage that may be, under some circumstances, about as large as the shrinkage due to air drying, at 70 F and 50 per cent relative humidity, from a saturated condition. Under these conditions some increase in weight will also take place [*20*]. Available evidence indicates that all of the constituents of cement are subject to ultimate carbonation under ideal conditions [*21*]. The rate and extent of carbonation are dependent on variables such as moisture content and density of member, temperature, concentration of carbon dioxide, time, size of member, method of curing, and the sequence of drying and carbonation. Carbonation proceeds slowly and usually produces little shrinkage at relative humidities below 25 per cent or near saturation. The maximum effect appears to occur when the relative humidity is around 50 per cent.

Less dense concrete products, such as building block made with lightweight aggregate, are more susceptible to carbonation than dense concrete products. Under normal conditions an increase in time, temperature, or concentration of carbon dioxide tends to increase carbonation shrinkage. Various factors that appear to be involved in the effect of specimen size include the paste porosity, the distance the water vapor released by carbonation must diffuse to reach a boundary, and the rate of water vapor and carbon dioxide diffusion. Concrete block cured in steam at atmospheric pressure obtains its maximum carbonation shrinkage in air at 50 per cent relative humidity, while autoclaved block are relatively immune to carbonation at that humidity level, but they experience carbonation shrinkage at higher relative humidities or under wetting and drying conditions [*22*]. In addition, it has been shown that precarbonation improved the volume stability of block cured in steam at atmospheric pressure, and that while precarbonation of autoclaved block eliminates the long-time carbonation shrinkage, it increases appreciably volume changes due to wetting and drying.

The possibility of improving the volume stability of concrete block cured in steam at atmospheric pressure and then subjected to drying and carbonation by hot-flue gases has been investigated [*23*]. Drying shrinkage of the finished product was reduced more than 30 per cent under favorable conditions. In order to obtain satisfactory reductions in shrinkage it was concluded that a 24 hr period of carbonation should normally be provided, but that under optimum conditions a shorter period may cause a considerable reduction in shrinkage. Kiln conditions suggested for effective precarbonation treatments included: relative humidity between 15 and 35 per cent, carbon dioxide content as high as possible but not less than 1.5 per cent, and temperature between 150 and 212 F.

Nonshrink or Expansive Concrete:

A concrete with low-drying shrinkage, known as Prepakt concrete, is made by filling the forms with graded coarse aggregate and then pumping a special cement grout into the forms until all voids are filled. After hardening the concrete has exceptionally low shrinkage because the coarse aggregate particles are in contact and are not separated by cement paste which causes most of the shrinkage in normal concrete. This type of concrete has had its greatest use in the repair of existing concrete structures.

Shrinkage may be minimized or prevented when concrete must be placed under special or difficult conditions such as bedments under machinery or around a congestion of reinforcing steel when vibration is not possible by the addition of small amounts of superfine unpolished aluminum powder or specified amounts of graded finely divided iron particles with added oxidizing and cement dispersing agents. These methods are used for limited special conditions and are not used for normal placement of structural concrete because of difficulties of proper control and possible resultant variable effects.

Attempts to formulate expansive cements that compensate for shrinkage by controlled expansion of cement have recently received additional attention [*24,25*]. Most of the recent work has involved cements in which the formation of hydrated calcium sulfoaluminates causes the expansive mechanism. Another use for the expansive cement is to induce stress in restraining steel. This type of cement is presently being used in the USSR for the production of self-stressed concrete pipe ranging between 6 to 40 in. in diameter. In the United States most of the work with expansive cements is still on an experimental basis. Recent examples of the use of this type of cement are two experimental "shrinkage-compensated" highway test sections in California. The chief difficulty associated with the use of expansive cements is the careful control required to obtain the desired expansion. The production of concrete with desired predetermined volume-change characteristics requires careful consideration and control of all factors involved in the production, with special attention to: (1) compositions, proportions, fineness of the components of the expansive cement; (2) proportions of the mixture; (3) time and temperature of the curing period; and (4) the percentage of steel used to restrain expansion.

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Hardened Concrete

THERMAL PROPERTIES

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The thermal properties of hardened concrete that are of engineering significance are thermal conductivity, specific heat, thermal diffusivity, and coefficient of thermal expansion. Thermal conductivity is the measure of rate of heat flow; specific heat is the measure of heat capacity; thermal diffusivity is an index of the facility with which the temperature can change; and coefficient of thermal expansion governs change in volume, or as usually measured, change in length with change in temperature.

Adiabatic temperature rise, or the heat of hydration of the cement, may well be considered as another, although transient, thermal property of the concrete. This property and proper control of it is of vital consideration in the design of modern, massive, concrete structures [*1–4*].[2]

Knowledge of these properties is used in designing and predicting the behavior of all types of structures, from the building of lightweight concrete in which insulation is a major factor to large massive structures in which artificial cooling may be employed. Stabilizing the structure thermally and volumetrically is of prime importance. An accurate knowledge of these properties or the ability to predict them with reasonable accuracy can do much to bring about better design and more efficient construction of all concrete structures.

Thermal Properties and Their Ranges

Thermal Conductivity:

The ability of hardened concrete to conduct or pass heat through itself is usually measured in terms of Btu ft/hr ft^2 deg F (kg cal m/hr m^2 deg C). This represents the uniform flow of heat in Btu per hour through a body of material 1 ft^2 in area with a temperature difference of 1 F/ft of thickness, or the uniform flow of heat in kilogram calories per hour through a body of material 1 m^2 in area with a temperature difference of 1 C/m of thickness. The range of this property is large, being from 0.8 to 2.5. Btu ft/hr ft^2 deg F for normal saturated concrete between 50 and 150 F, or from 1.19 to 3.72 kg cal m/hr m^2 deg C between 10 and 65 C, and is principally dependent upon the composition of the concrete [*5*]. Although conductivity of lightweight concrete varies with its density, conductivity of normal to heavyweight concrete seems to be nearly independent of density. For instance, concrete made with barite aggregate has a conductivity of 0.8 Btu ft/hr ft^2 deg F (1.19 kg cal m/hr m^2 deg C) with a density of 227 lb/ft^3 (3636 kg/m^3), Bull Run Dam concrete (gravel largely igneous) 0.83 Btu ft/hr ft^2 deg F (1.24 kg

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

cal m/hr m^2 deg C)[3] at 159 lb/ft^3 (2547 kg/m^3), and Norris Dam concrete (crushed dolomite coarse and fine aggregate) 2.13 Btu ft/hr ft^2 deg F (3.17 kg cal m/hr m^2 deg C) at 160 lb/ft^3 (2563 kg/m^3). Conductivity of lightweight concrete varies from 0.08 Btu ft/hr ft^2 deg F (0.119 kg cal m/hr m^2 deg C) to 0.42 Btu ft/hr ft^2 deg F (0.51 kg cal m/hr m^2 deg C) for densities from 30 lb/ft^3 (480 kg/m^3) to 110 lb/ft^3 (1762 kg/m^3) ovendried. The effect of temperature upon conductivity is usually relatively insignificant, and the change for an increase in temperature range is likely to be positive for low-conductivity concrete and negative for high-conductivity concrete.

Prediction of thermal conductivity of concrete from the composition is likely to be uncertain as well as a major problem due to the many factors involved. Each mineral present in the aggregate must be identified and assigned its share of effect. Both air content, or void content, and water contained or included tend to reduce the thermal conductivity, except that thermal conductivity of concrete whose unit weight is less than 100 lb/ft^3 is increased by increased water content. Although drying reduces the amount of water, a low-conductivity ingredient in the concrete, it further lowers conductivity because this loss increases air voids of still lower conductivity. The air content of concrete, whether original or as air voids due to drying, has a pronounced effect in reducing the conductivity of the mass. The conductivity of hydrated portland cement paste 0.8 Btu ft/hr ft^2 deg F (1.19 kg cal m/hr m^2 deg C), although high compared to that of water 0.35 Btu ft/hr ft^2 deg F (0.5 kg cal m/hr m^2 deg C), is still relatively low and tends to be one of the factors reducing the thermal conductivity. Basalt and trachyte are among types of rock used as aggregates having low conductivity and tending to reduce the conductivity of the mass [5]. Quartz, on the other hand, is an extremely high-conductivity constituent, and various investigators have indicated its k value as being 7.0 Btu ft/hr ft^2 deg F (10.42 kg cal m/hr m^2 deg C) parallel to the axis of the crystals and above 4.0 Btu ft/hr ft^2 deg F (5.95 kg cal m/hr m^2 deg C) at right angles to this direction. Dolomite and limestone have conductivities near the usual upper limit of hardened concrete. Lightweight aggregates have lower conductivities, usually in the same order as their densities.

Specific Heat:

The amount of heat necessary to change the temperature of 1 lb of material 1 F (1 g of material 1 C) is defined as specific heat, which for hardened concrete varies from 0.20 to 0.28 Btu/1 lb/deg F (cal/g/deg C). The effect of mineral composition of solids constituents per pound upon specific heat is relatively insignificant when compared with that of the water. In general, the specific heat varies directly with variation in the temperature [5]. Hydrated cement paste has a low specific heat and a very definite curve of variation with temperature. Since water has a specific heat of 1.0, the water content is relatively effective in raising the specific heat of concrete. The specific heat of ice at 0 F is 0.48. That of most other nonmetallic construction materials is between 0.16 and 0.24.

Thermal Diffusivity:

The so-called thermal diffusivity "constant" is a measure of the rate at which temperature changes will take place within the mass of hardened concrete. Its variability over a range of from

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[3] Figures in parentheses are approximate metric equivalents.

0.020 to 0.080 ft^2/hr (0.00186 to 0.0074 m^2/hr) [*5*] is controlled largely by the composition of the mass and is very similar in characteristics to the thermal conductivity. This property is directly proportional to the thermal conductivity and inversely proportional to the specific heat multiplied by the density. It may be determined from the formula:

$$a = \frac{k}{c\rho}$$

where:

a = thermal diffusivity (diffusion constant),
k = thermal conductivity,
c = specific heat, and
ρ = density in pounds per cubic foot.

Coefficient of Thermal Expansion:

All materials exhibit a change in dimension upon temperature change. In engineering materials, this property is usually positive—that is, producing increased length at increased temperatures—and is frequently considered to be a constant. It is expressed in terms of inches per inch per deg F (millionths per deg F) (millionths per deg C) and, for hardened concrete, is usually between $3\frac{1}{2}$ and $6\frac{1}{2}$ millionths per deg F (6.3 to 11.7 millionths per deg C). When a precise value is not required, a value of 5 or 5.5 millionths per deg F (9.0 or 9.9 millionths per deg C) is frequently used. The composition of concrete completely controls the coefficient of thermal expansion of the hardened mass, provided that the variability of thermal coefficient attending moisture changes is taken into account.

Research by the author [*6*] and by Meyers [*7*] shows that moisture content may cause the coefficient of expansion of neat cement to vary by as much as 100 per cent, having minimum values in either ovendry or saturated conditions and maximum values somewhere in the region of 70 per cent saturation. Meyers [*7*] found that either autoclaving or aging for several years reduced the thermal coefficient of neat cement at intermediate moisture contents. The thermal coefficient for neat cement also depends upon the particular cement [*6*] and may possibly vary from less than 5 to more than 12 millionths per deg F (9.0 to 21.6 millionths per deg C) depending upon differences in cement and the moisture content. Tests of concrete made with some manufactured lightweight aggregates indicate that the thermal coefficient of expansion is much lower, 3.4 millionths per deg F (6.1 millionths per deg C), than that of concrete containing natural sand and gravel. The higher thermal coefficients of concrete at intermediate moisture content may be a function of both the composition of the cement and properties of the aggregate. Recent tests show that some natural aggregate concretes, including concrete at Shasta Dam, California, have a coefficient of thermal expansion that varies greatly with moisture content, whereas other normal weight and lightweight concretes exhibit nearly constant thermal coefficient of expansion for all moisture conditions. The thermal coefficient of expansion of cement pastes usually varies greatly with moisture content [*6*].

Since the expansion of hardened concrete will be equal to the composite or weighted average expansion of its constituents [*6,8*], the thermal coefficients of various mortar mixes should be expected to vary from about 5 millionths to about 8 millionths per deg F (9.0 to 14.4 millionths per deg C). The thermal expansions of various American rocks that may be used as aggregate vary from less than 2 to more than 7 millionths per deg F (3.6 to 12.6 millionths per deg C) [*6,9*]. Some of the materials, such as feldspar and quartz which have high thermal coefficients or bedded materials such as limestone and sandstone, show anisotropic characteristics.

Investigations indicate the thermal coefficient of expansion to be essentially constant over the range from 15 to 70 F (−9.46 to 21.1 C) in dry concrete. However, moist concrete, especially that which is nearly saturated, frequently shows a significant increase in thermal coefficients with increasing temperature.

Adiabatic Temperature Rise:

The property of adiabatic temperature rise is a function of the proportion and specific heat of the various mix constituents and the heat of hydration of the cement used. Unlike the usually considered thermal properties, which are relatively fixed by the aggregate available, this property may be controlled over an appreciable range. Mix proportions, type of cement, and pozzolanic materials are effective means through which adiabatic temperature rise may be controlled [*2–4*].

Laboratory tests [*10*] show mass concrete mixes of a few years ago which contained approximately four sacks of cement per cubic yard to have adiabatic temperature rise of from 47 F (8.3 C) for low-heat cement to about 70 F (21 C) for standard Type I cement. The latest practice using Type II cements with pozzolans and efficient water-reducing agents employ mixes as lean as two sacks of cement and one of pozzolan, and thus limit adiabatic temperature rise to not more than 36 to 40 F [*11*]. Leaner mixes also produce less temperature rise in proportion to the cement used. The benefit of water-reducing agents lies principally in allowing workable mixes with satisfactory strength to be produced with less cement. Tests indicate that cements manufactured since World War II have approximately 10 per cent greater heat of hydration than did others of the same type manufactured previously. This change is at least in part the result of factors other than fineness of grinding. Fineness increases the rate of heat generation at early periods, but the ultimate heat of hydration is increased only slightly.

Pozzolanic materials usually produce much less temperature rise than the cement which they replace. The temperature rise produced by pozzolanic materials is ordinarily slower and occurs at a later, more favorable time. This is especially so with fly ash. Sometimes pozzolans are used for economy and to inhibit alkali-aggregate reaction, as well as to control temperature rise. In modern mass concrete construction, it is not unusual to produce lean mixes with Type II cement, pozzolanic materials, and retarders, which have acceptable strength and adiabatic temperature rise of 40 F or less.

SIGNIFICANCE OF THERMAL PROPERTIES IN MASSIVE STRUCTURES

Stresses and Cracking:

It is obvious that a large massive concrete structure, such as a gravity dam, presents a problem involving stresses due to shrinkage and thermal action. Such structures should upon final completion be monolithic in intimate contact with the foundation and abutment in order that stress distribution and limiting stresses be as designed. Zienkiewicz [*10*] emphasized the need for knowledge and consideration of thermal properties in controlling stresses in massive concrete structures. Blanks, Meissner, and Rawhouser [*1*] analyzed crack surveys and design features of several large dams and concluded that thermal stresses are the principal cause of cracking in massive structures.

Rawhouser [*2*] and Townsend [*3,4*]

explain design methods which eliminate cracking and control joint openings by controlling temperature in mass concrete. These papers show that thermal shrinkage which causes cracks can largely be controlled by limiting the amount of temperature rise above the final stable temperature.

A complete knowledge of the thermal properties is necessary in designing the mix and block size and in outlining the cooling and construction procedures. Proper design can limit temperatures and resultant stresses to safe values.

Whereas the design of such dams as Hoover and Grand Coulee called for construction in blocks 50 by 50 ft (15.24 by 15.24 m) max size, in 5-ft (1.52-m) lifts, modern concrete construction now permits blocks as large as 50 by 150 ft (15.24 by 45.72 m) or slightly larger when construction is sufficiently massive and 7.5-ft (2.29-m) lifts. Blocks of this size are permissible because precooling of materials to limit initial temperatures and concrete mix design and control technology to limit temperature rises can prevent the necessity for excessive cooling which would cause unsafe tensile stresses.

Concrete placing temperature is frequently limited, as in Glen Canyon Dam, to 50 F (10 C), and the permissible temperature rise in this case was successfully limited to 25 F (18 C), thus permitting maximum size blocks.

Cooling and Grouting of Dams:

The heat of hydration of cement compounds during setting produces a very appreciable temperature rise in large massive structures from which heat cannot readily escape. The temperature difference above the mean annual temperature must be relieved in order for the mass to be stable and capable of bearing the waterload. Usually positive measures are in order, both for controlling the amount and rate of heat generation and for dissipation of heat [*2–4*]. Frequently, as in the case of Hoover Dam, artificial cooling installations are provided. In many cases the artificial cooling can be accomplished by means of circulating river water at ambient temperatures in the cooling pipes located between each successive lift of new concrete. The amount of heat to be removed and the thermal properties of the concrete control the design of the cooling system and the height of the placement lifts. Modern lean mixes with low temperature rise have in addition to allowing larger blocks permitted an increase in depth of concrete lifts from 5 ft (1.52 m) to 7½ ft (2.29 m).

In some cases, such as Glen Canyon Dam, precooling of materials by refrigeration was necessary to provide placing temperatures which would keep final temperatures within acceptable limits. In other cases, only a slight amount of precooling was necessary, and this was accomplished by sprinkling the aggregate during hottest weather. In any case, when grouting is necessary to secure monolithic action, steps must be taken to provide that a large massive concrete structure comes to its final temperature and stable dimensions during or at the end of the construction period in order that grouting may make a final approach to a monolithic structure.

When no artificial cooling methods are provided, thermal properties of concrete are important because construction schedules and formed blocks must be so arranged that heat dissipation will take place advantageously. These properties must be known to the designer for arranging block sizes and specifying the rate of concrete placement. Current specifications for precooling procedures, placing temperatures, and rates are based mainly upon the use of heat of hydration, specific heat, thermal conductivity, and thermal diffusivity values. Large temperature changes after completion cannot be tolerated because large tensile stresses and cracks will result [*1–4*].

Where artificial cooling means are to be provided, thermal diffusivity is the controlling factor in the spacing of cooling pipes within the mass. Data of thermal conductivity and specific heat along with diffusivity are used to determine the size of cooling pipes and rates of cooling-fluid circulation, thus determining the size of the refrigeration plant, if any, or the amount of water to be circulated. The structure must be so designed that thermal shrinkage will open contraction joints by an amount that may be readily and surely grouted.

While laboratory installations have successfully grouted simulated joint openings as small as 0.010 in. (0.25 mm), it is not practical to attempt grouting such small openings in a massive structure.

Although structural openings smaller than 0.04 in. (1.02 mm) have been successfully grouted in some cases, this is usually considered to be a minimum for assured success. The Bureau of Reclamation designers normally plan for an average joint opening where grouting will be required of at least 0.08 in. (2.03 mm) [*3,4*] in order to assure that unusual circumstances will not make some joints too small.

Where structures are sufficiently massive and ambient temperatures will permit, the concrete is normally cooled slightly below mean annual temperature prior to the grouting operation in order that permanent joint closure can be assured by moderate compressive stresses.

SIGNIFICANCE OF THERMAL PROPERTIES IN OTHER LARGE CONSTRUCTION

Arches and Large Structures:

The design and construction of arches and other continuous concrete structures, when concrete thickness is only a few feet, do not usually require consideration of thermal properties other than the coefficient of thermal expansion. Coefficient of expansion must be considered in all such structures if they are to resist cracking and function as continuous structures. Where expansion joints cannot be provided at suitable intervals, thermal expansion must be converted to equivalent stress and adequate provisions made for this additional load. Conductivity, specific heat, and diffusivity are important to such structures only as these properties affect durability by their tendency to accentuate local strain within the mass caused by daily temperature cycles. Weiner [*13*] considered durability of exposed concrete to be a function of diffusivity and thermal expansion.

Highways:

The principal thermal property to be considered in highway design is coefficient of thermal expansion. The location of expansion joints is determined by the amount of thermal expansion and by the strength and cross section of the highway slab. Other thermal properties are considered in highway work as they affect durability of concrete either through thermal strain or incompatibility [*14,15*] or, to a lesser degree, as they may affect the tendency of a highway slab to curl slightly from day to night and be broken at the corners by heavy loads. This latter situation, however, is usually considered from the structural standpoint only and not from the standpoint of the thermal properties that produce it.

Winter Concreting:

A knowledge of the thermal properties of concrete has made possible the designs now used in many cases for winter concrete work, where insulated forms provide the necessary protection from freezing. Thermal diffusivity, conductivity, and heat of hydration were used to compute the insulation thickness necessary to protect winter concrete from freezing, especially at outside corners. Sometimes calcium chloride is used as an accelerating admixture when mean ambient temperatures go below 40 F (4.4 C). This acceleration aids somewhat in winter protection by increasing the rate of early heat generation and shortening the necessary duration of protection.

SIGNIFICANCE OF THERMAL PROPERTIES TO BUILDING CONSTRUCTION

Thermal Conductivity:

Few buildings, residential or industrial, are now designed without consideration for thermal insulation. Normally, concrete would not be considered to be an insulating material, but special concretes have been entering the field of building construction where insulation is required. Many lightweight aggregates and cellular concretes are now available which provide some degree of insulation, in addition to having low unit weight, hence, being more desirable from the standpoint of dead load in structural uses. Several concretes have been tested as insulation materials for housing purposes [*16,17*]. Conductivity of lightweight concrete may vary from 0.08 to 0.35 Btu ft/hr ft^2 deg F (0.119 to 0.52 kg cal m/hr m^2 deg C) when dry.

Thermal Expansion:

Thermal properties of concrete, other than conductivity, are not usually considered in building construction, except as the coefficients of expansion affect stresses and expansion joints. Frequently, a structure is too small for this consideration to be a major item, although the modern tendency toward continuous structures requires a knowledge of the coefficient of thermal expansion in their design.

Thermal expansion also becomes a problem in some cases for concrete repair where epoxy mortars are used either in repairing construction deficiencies or deteriorated areas. Epoxy resins, in general, have thermal coefficients well above 20 millionths per deg F (36 millionths per deg C), and well-designed epoxy mortars may have coefficients of 13 millionths per deg F (23 millionths per deg C). This latter value, 2½ times the normal for portland cement concrete, can give rise to thermal incompatabilities and possible failures where large applications are subjected to extreme temperature changes in cold climates.

METHODS OF TESTING

Conductivity:

Concrete for massive structures is naturally tested in the wet- or moist-cured condition as it will be used. Some laboratories prefer to determine conductivity as a direct test. Reference [5] describes the method used in the Bureau of Reclamation laboratories. Other laboratories have determined this property by computation from the specific heat, density, and diffusivity which they determine by direct tests.

Concrete for building construction, especially insulating concretes, may be tested by a method similar to that used for other insulating materials (ASTM Test for Thermal Conductivity of Materials by means of the Guarded Hot Plate and Test for Thermal Conductivity and Transmittance of Built-Up Sections by Means of the Guarded Hot Box (C 177 or C 236), respectively). Many methods of test are available.

The American Society of Heating, Refrigerating, and Air-conditioning Engineers (ASHRAE), *Guide and Data*

Book [20], or other current sources available at good libraries, should be consulted for additional data and latest details when thermal properties of special concrete used in building construction are being considered. Current files covering latest year of such periodicals as *Air-Conditioning, Heating, and Ventilating* by Industrial Press, New York, N. Y., and books such as *Handbook of Air-Conditioning, Heating, and Ventilating* [21], or similar books available in large libraries, frequently give recently developed data, including the latest in materials processes.

Specific Heat:

Specific heat of concrete is frequently determined by a simple "method of mixtures" process. The principal piece of equipment usually consists of a high-insulation-type calorimeter large enough to accommodate the size sample deemed necessary for representative results.

The *Procedures Manual* of the Bureau of Reclamation [18] describes the form of test for approximately 70-lb samples. The Corps of Engineers' *Handbook for Concrete and Cement* [19], under Designation CRD-C-124-62, describes a test for samples up to 2 lb in size.

Diffusivity:

Diffusivity may be computed from the other thermal properties, provided they are determined. Some laboratories prefer to make direct tests of thermal conductivity, specific heat, and density and compute the thermal diffusivity, whereas other laboratories [19], Designation CRD-C-36-63, prefer to make direct tests for diffusivity and the compute conductivity. When diffusivity is measured directly, the test consists essentially of a rate-of-cooling determination with measurements being taken at the center of a mass of concrete which has been previously tempered to a high constant temperature and then cooled in a constant water bath.

Coefficient of Thermal Expansion:

Methods of measuring the coefficient of thermal expansion are almost as numerous as the number of laboratories making such measurements. They vary from tests on small specimens held in extensometer frames [6] or small specimens with attached strain gages for which the temperature is controlled in an oven, to large bars tested in horizontal comparitors [18]. Larger laboratory specimens or field structures may be tested with Whittemore-type strain gages or embedded Carlson strain gages in some instances.

Temperature Rise and Heat of Hydration:

Both temperature rise and heat of hydration may be determined by adiabatic calorimeter methods [17]. The heat of hydration of portland cement may also be determined by heat of solution methods such as ASTM Test for Heat of Hydration of Portland Cement (Short Method) (C 186) or Federal Test Method Standard 158.

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Hardened Concrete

PORE STRUCTURE

By George Verbeck[1]

Most of the important properties of hardened concrete are related to the quantity and the characteristics of the various types of pores in the paste and aggregate components of the concrete. The engineering properties of concrete, such as strength, durability, shrinkage, and permeability, are directly influenced or controlled by the pores in the concrete and the relative amounts of the different types and sizes of these pores.

The pores can exert their influence on the properties of the concrete in various ways. As regards the strength and elasticity of the concrete, it is primarily the total volume of the pores that is important, not their size or continuity. The permeability is influenced by the volume, size, and the continuity of the pores. Shrinkage, at least that part of drying shrinkage that is reversible, is largely a function of changes in surface energy at the solid to pore interface and, therefore, depends upon the nature of the solid surface and the total surface area of the pore system. Irreversible drying shrinkage may involve capillary phenomena. The resistance of concrete to freezing and thawing and deicer scaling is controlled by the volume and the characteristics of entrained air voids. It is, therefore, not surprising that there has been considerable interest in developing ways of measuring and characterizing the different types of pores in concrete and in elucidating the various mechanism by which they influence the properties of the concrete.

In order to understand the significance of the various pore types and their characteristic effects and interrelationships in connection with some of these engineering properties, it is important to understand the manner in which these pores originate, the factors that affect them, and the limits within which their quantities may vary.

Porosity of Concrete

The pores formed in the original plastic concrete are either water- or gas-filled. After the concrete has hardened, the water pores may tend to dry, or the air pores may tend to become water saturated, depending upon the history of the concrete, the external moisture conditions, and the dimensions of the concrete member.

Concrete is made up primarily of cement paste and aggregate components; in addition, it contains a void component composed of air voids, "under-aggregate" fissures, and, on occasion, "honeycomb" pockets. "Honeycomb" can be considered to be an abnormal and undesirable condition that can be avoided by use of proper mixture proportions and placing practice and, therefore, needs no discussion for the present purpose.

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Fissures or zones of weakness may at times develop under aggregates during the period of bleeding. Little is known concerning the frequency with which they occur, except in extreme cases. Presumably their occurrence is a function of the water content of the concrete, the bleeding properties of the cement, and the aggregate grading (the amount of fine material and the bridging of coarse aggregate). They may be difficult to detect, since the phenomena may produce only a weakened zone of high water-cement ratio (having almost normal appearance) immediately under pieces of aggregate.

Capillary and Gel Pores in Cement-Water Pastes:

The water-filled space in a freshly mixed neat cement paste represents space that is available for the formation of cement hydration products. As hydration proceeds, the volume of this space, which initially was determined by the water-cement ratio of the paste, is continually reduced by the formation of the hydrated "gel" which has a bulk volume larger than the original unhydrated cement itself. At any time, that part of the original water space which has not become filled with hydration products constitutes the "capillary" system of the paste. It can thus be seen that hydration reduces the size and volume of the capillary space in the paste. If the original capillary space is low (water-cement ratio about 0.35 by weight), the bulk volume of the gel eventually will be sufficient to fill this space and produce a paste free from capillary space. At higher water-cement ratios, the gel volume is not sufficient to fill completely all the original water space in the paste, even after complete cement hydration, and pastes having relatively large volume of capillaries may be produced. This capillary system can be visualized as a submicroscopic system of voids randomly distributed throughout the hydrated cement paste matrix. These submicroscopic capillary spaces include a wide variety of "sizes" and "shapes" but perhaps can be considered to vary from about 0.000008 to 0.013 mm in diameter, the range depending upon the original water-cement ratio and the degree of hydration of the cement. Such estimates of size of capillary spaces and gel pores to be discussed later are based on the assumption of a cylindrical pore shape and on calculations of the "hydraulic radius" of the pore system, this being obtained by dividing the volume of the system by its surface area. For cylindrical pores the hydraulic radius is one fourth of the diameter, and for slit-shaped or plate-like pores (now considered most typical of the gel pores) the hydraulic radius is one half of the width. These capillary spaces may or may not be directly interconnected, as will be discussed subsequently.

Considering the cement paste component (which does not include air voids), even a dense, well-cured paste is capable of holding relatively large quantities of evaporable water, although no capillary spaces are present. The hydration product or gel with which the original capillary spaces within the paste are filled is itself porous, containing pores that are smaller than the capillary pores. These pores have been referred to as "gel" pores. Water in these small pores (they vary from perhaps 0.000001 to 0.000008 mm in mean diameter) will have characteristics, such as vapor pressure, freezing characteristics, and mobility, notably different from those of free water in bulk. This is also true, but to a lesser degree, for water in the larger capillary spaces. The volume of the gel pores in each quantity of paste increases with continued hydration of the cement, whereas the volume of the capillary spaces decreases with hydration.

From recent studies of pore size distribution of nitrogen adsorption it appears that there is no unique single size for these gel pores nor that the gel pores are totally different in size than some of the capillary pores. Although there appears to be a broad and continuous spectrum of sizes in the gel-capillary pore system, the two systems can be somewhat arbitrarily separated at an average calculated "pore" diameter of about 0.000008 mm. Numerically, most of the capillary pores appear to approach this small size. However, each gross unit volume of hydration product, exclusive of the large capillary spaces, has commonly been considered to contain a characteristic volume of these small gel pores, about 25 to 28 per cent by volume, the exact porosity apparently varying somewhat with cement composition.

In the broad aspect, the concepts regarding nature of the pores in hydrated paste just discussed are believed to be essentially correct. However, recent measurements of pore size distributions in hydrated pastes, using nitrogen and water vapor techniques and electron micrographic examination of replicas of paste surfaces, indicate that changes in details of these concepts will perhaps be required. Unfortunately, these studies have not advanced as yet to the stage at which the anticipated changes in concept of the pore structure can be validly defined.

The product of cement hydration that constitutes the solid fraction of the hydrated cement gel is composed of very fine particles of matter. A calcium silicate hydrate ("tobermorite gel") may constitute about 50 per cent of the cement gel. It is a poorly crystalline material, consisting of small, extremely thin sheets that may be crumpled or rolled as foils. Another product of the hydration of the calcium silicates is calcium hydroxide, which may form crystals large enough to be visible in an optical microscope and which may constitute 25 per cent of the cement gel. The over-all average particle size of the elements of hydration gel is so small that their specific surface area is in the vicinity of 175 to 200 m^2/g of dry paste. If the hydration gel were to consist of spherical particles of equal diameters, the spheres would have a diameter of only about 140 A (0.000014 mm).

As previously noted, water held in either the gel or capillary pores does not behave as normal free water. The gel pores retain significant quantities of surface adsorbed water and chemisorbed or hydrate water or both, even at relatively low humidities. Because of the larger size of the capillary spaces, proportionately less of the capillary water is strongly adsorbed on the pore walls, and it is therefore relatively more volatile than is gel water; the water in the capillary spaces is almost completely evaporable at humidities below about 40 per cent. Above 40 per cent relative humidity "capillary condensation" occurs, that is, the capillaries become filled with water by precipitation from the vapor phase.

It is the capillary spaces with which the permeability of paste and concrete is most closely associated, for the water in these pores can move much more freely under hydrostatic pressure than can the adsorbed gel water.

Aggregate Pores:

Aggregate constitutes about 75 per cent of the volume of concrete. Consequently, the properties of the concrete are significantly influenced by the characteristics of the aggregate. The porosity of common aggregate materials ranges from nearly 0 to as much as 20 per cent by solid volume. For lightweight aggregates the porosity may be in the range of 30 to 50 per cent. The size of the pores also varies greatly among different aggregates. In very dense aggregates, such as granite, marble, or some traprocks, they are in the size range of paste capillary spaces of intermediate size. Frequently they are at least the size of the largest paste capillaries. Some aggregates, such as certain sandstones and lightweight aggregates, have a very coarse pore system. In some instances the distribution of pore sizes can be determined by microscopical techniques or estimated by use of a mercury porosimeter. As in the case of the cement paste pores, the size distribution of aggregate pores also can be estimated using nitrogen or water vapor adsorption methods. The total volume and the size distribution of the pores in aggregates have an important influence on the volume stability and particularly the frost resistance of the aggregates and the concrete.

Air Voids (pores):

Concrete normally contains air voids, accidentally entrapped or purposely entrained, dispersed throughout the paste component. Many of the accidentally entrapped voids can be seen with the unaided eye and may range in size up to about 2 mm (0.1 in.). Those purposely entrained voids that have a significant effect on the resistance of the concrete to freezing and thawing and deicer scaling are in the range of 0.025 to 0.050 mm (0.001 to 0.002 in.) in diameter. The air voids may constitute from less than 1 to more than 10 per cent of the concrete volume, the volume and size depending upon several factors including the amount of air-entraining agent used, size distribution and maximum size of aggregate, concrete consistency, duration of mixing, and so on.

The small air voids having a significant effect on concrete durability can be seen and studied using a microscope and a magnification in the range of approximately 30 to 150 diameters. The volume of the air voids in hardened concrete and the characteristics of the air-void system (void size, voids per inch of traverse and "spacing factor") can be determined in accordance with ASTM Recommended Practice for Microscopical Determination of Air-Void Content, Specific Surface, and Spacing Factor of the Air-Void System in Hardened Concrete (C 457 – 60 T).

Pores and Voids in Concrete:

In summary, concrete may thus be visualized as consisting of a heterogeneous mixture of components, each component having its own characteristic pores. In terms of the other pores in the concrete, the air voids, normally the coarsest of all, may constitute from less than 1 to more than 10 per cent of the total volume of the concrete. Approximately 75 per cent of the concrete is aggregate, frequently heterogeneous, with an internal pore volume varying from almost 0 to 20 per cent or more (most commonly about 1 to 5 per cent), the pores ranging from relatively fine to coarse. The cement paste component usually contains both extremely fine gel pores and the coarser but submicroscopic capillary spaces. The gel pores are currently considered to constitute about 28 per cent of the paste volume, and the capillary spaces an almost negligible volume for an essentially fully hydrated paste having an initial water-cement ratio in the range of 0.35 to 0.38 by weight. A fully hydrated paste of 0.70 water-cement ratio would contain about 20 per cent gel pores and 30 per cent capillary spaces by volume. Incomplete hydration would decrease the volume of gel pores and increase the volume of capillary spaces proportionately.

Although these various pores and voids in concrete influence the physical properties of water contained therein, the properties of the separate types of pores are not sufficiently different to permit their complete identification in concrete. The combined porosity of concrete (including the air voids) can be represented by the total capacity for evaporable water between the stages of complete saturation and dryness—dry except for the combined or nonevaporable water content of the cement hydrate.

PERMEABILITY OF CONCRETE

Interest in the permeability of concrete arises usually in connection with some proposed specific application of the concrete and, therefore, may result in emphasis of some particular aspect of concrete permeability.

Movement of water through concrete can be produced by various combinations of air or water pressure differentials, humidity differentials, and solutions of different concentrations (osmotic effects) or at different temperatures. Although it is known that the observed rate of movement is dependent upon the characteristics of the membrane and the permeating material, much more needs to be learned regarding these relationships. Various tests have been devised to determine permeability. Although these procedures may reveal the relative characteristics of the concretes involved, the quantitative value obtained may depend considerably upon details of the experimental conditions.

For example, considering the relatively simple case of a hydrostatic water pressure differential across a membrane of concrete, it has been observed that many factors may influence the rate of flow of water through the membrane. The prior curing history of concrete, the direction of permeation in relation to casting position (under-aggregate fissures), the treatment of the surfaces (as-cast, sawed, or sand-blasted), carbonation, the downstream conditions (whether air or water), the measurement of inlet or outlet flow or both, the nature and amount of solutes in water, and the electrical conductivity between the membrane faces are factors that must be considered. However, the test procedures commonly used for determination of the water permeability of concrete are probably sufficient, in most cases, to reveal relative differences in the permeability of concretes made with different water contents, admixtures, and so on.

At the present time the qualitative effects of many factors on the water permeability of concrete are known. The permeability of concrete to water under hydrostatic pressure will depend significantly upon the permeability of the cement paste component of the concrete, providing the concrete is intact—not previously damaged by frost or rapid drying and not containing excessive under-aggregate fissures or honeycomb. All of the permeating water must pass through the paste component of the concrete (the continuous phase), and if the paste is of low permeability, the concrete will show similar characteristics.

The hydrostatic water permeability (expressible in terms of Darcy's law) of a well-cured paste is reduced approximately a thousandfold by reduction in water-cement ratios from 0.8 to 0.4 by weight. This large reduction in permeability is due to the drastic reduction in capillary size and volume that accompanies the decrease in water-cement ratio. All of the capillary volume in fresh paste is capable of rapid transmission of water (as revealed by bleeding); this "permeability" rapidly decreases with hydration. The permeability of a paste of 0.8 water-cement ratio may decrease a thousandfold between the curing ages of 7 days and 1 year. Thus, there is a millionfold difference between the permeability of high-water-ratio paste at early age and that of well-cured low-water-ratio paste.

The permeability of cement paste appears to undergo a relatively abrupt reduction when, because of original water-cement ratio and extent of cement hydration, it attains the condition at which the capillary system (which has the greatest influence on permeability) becomes discontinuous due to segmentation and blocking by increase in the concentration of the gel component in the paste. This attainment of discontinuity in the capillary system appears to occur when the total porosity (gel plus capillary) reaches approximately 50 per cent, the exact porosity required depending upon the fineness of the original cement. It has been estimated that cement pastes of 0.40, 0.50, 0.60, and 0.70 water-cement ratio require approximately 3 days, 14 days, 6 months, and 1 year of normal hydration, respectively, in order to attain this condition. There is evidence that even when the capillary system becomes "discontinuous" (as measured by water permeability) there still remains some continuity in the large pore system. This seems necessary in order to account for the spontaneous propagation of freezing in saturated pastes. Apparently the large capillary spaces necessary for the propagation of ice formation are not sufficiently numerous to influence significantly the permeability measurements.

The permeability of particular dense, impervious aggregates, such as traprock or marble, has been observed to be as low as that of mature pastes of about 0.40 water-cement ratio. Some granites have a permeability comparable to that of a mature paste of 0.70 water-cement ratio, despite the enormous differences in porosity, namely, about 50 per cent for the 0.70 water-cement ratio paste and about 1 per cent for the granite. Obviously, if the aggregate is more or less permeable than the paste matrix, the permeability of concrete will be changed accordingly but probably not greatly.

Significant quantities of air voids in the concrete, because they are much larger than the capillary and gel pores and, hence, do not impede movement of water, should increase the permeability of saturated concrete roughly in proportion to their quantity, provided other factors remain constant. However, other factors seldom do remain constant—it is commonly observed that air entrainment in most practical concretes will reduce segregation and bleeding and permit reductions in the water-cement ratio—with the result that the concrete may actually be more impermeable despite the presence of the air voids.

Capillarity can also produce liquid movement in concrete. The presence of water at one face of the concrete and unsaturated air at the other may give rise at the air interface to large negative pressures or capillary tensions (meniscus effects) which tend to draw the water through the concrete. Movement is produced by any moisture difference across the membrane. Air with different moisture content at the two surfaces will produce vapor diffusion through the pores in the concrete and along the gel surfaces. Osmotic pressure differentials can also produce movement comparable to hydrostatic differentials. Temperature and electrical forces may also be involved.

The numerous pores in concrete are normally lined with water—relatively immobile water—adsorbed on the pore walls. Presumably the pore area available for the relatively free flow of fluid is greatly reduced by this immobile lining, particularly in the small pores where it may constitute most or all of the cross-sectional area of the pore. However, when concrete is air-dried, the coarser capillary spaces are completely emptied of water, and the finer capillary spaces and gel pores partially emptied due to decreased water adsorption on the pore walls. This results in a drastically increased transmission area available for the movement of air or other gas. Pastes and concretes that have been dried are very permeable to air, thousands of times more permeable than they are to water.

ABSORPTION OF CONCRETE

The term "absorption" is usually applied to concrete in regard to the weight gain of partially dried specimens upon contact with or immersion in water. In practice, owing to the long periods of time required to establish equilibrium moisture conditions, measurement of weight gain is made from some semidry, nonuniform moisture condition to a condition approaching saturation.

During absorption it may be considered that the larger paste capillary spaces are the first to be wetted, with the finer gel pores perhaps next, their rate of "absorption" (primarily surface adsorption) being slow because of their low water permeability (low surface diffusion). The coarse pores in aggregate can become nearly filled with water only after a relatively high degree of saturation (and water vapor pressure) is established in the paste surrounding the aggregate. Because of its usually much finer pore structure, the paste, if it is much below saturation, can easily remove most of the aggregate water. Upon very long-continued exposure to water, the air voids, particularly the smaller voids, may become filled with water. Such a process requires that the air in the void compressed by the absorption process must dissolve in the capillary water and slowly diffuse out of the specimen to permit filling of the void with liquid.

The results of the absorption tests depend upon the procedures used. The absorption observed will depend obviously upon the initial state and uniformity of dryness of the concrete. Absorption occurs very quickly just after immersion of the concrete, but the rate decreases rapidly with time. Considerable time is required to reach apparent saturation equilibrium, the identification of equilibrium being clouded by the normal water gain of concrete accompanying continued hydration, osmotic effects, and leaching. Factors significantly influencing absorption are the curing history, water-cement ratio, aggregate characteristics, air content, cement type and fineness (particularly at early ages), specimen size and shape, method of surface preparation (cast, broken) surface carbonation, and so on.

The absorption test is of value primarily as a basis of comparison of different concretes, the absorption in a gross manner being a function of the permeability and porosity of the specimen although influenced by many factors of test procedure. With an appreciation of the many factors influencing absorption tests, both the rate and amount of absorption, it is not surprising that only rough correlations are generally observed between the results and such factors as water-cement ratio, strength, durability (freezable water content), and attack by aggressive solutions.

PORE-WATER PRESSURES

There are several mechanisms by which pressures can be developed in the pores of concrete. These pressures can be either positive (liquid in compression) or negative (liquid in tension). The positive pressures are of greatest importance since they are usually associated with undesirable effects in the concrete—freezing stresses, alkali-silica reaction, strength reductions, or uplift in dams. Tests of pore-water pressure *per se* perhaps have not been developed sufficiently to be of general immediate concern, although the gross effects of these pressures influence results obtained from other tests of the performance of concrete. A brief description of these pressures may suffice for the present purpose.

Freezing Pressure:

When the concrete is frozen, most of the water in the paste capillaries and the aggregate will freeze at temperatures somewhat below the normal freezing point. This freezing is accompanied by an expansion of the water-ice system, and if the concrete is critically saturated or nearly so, this excess volume will tend to be expelled from the capillary spaces into the air voids in the concrete. The pore pressures that are developed during this process will depend upon the amount of freezable water, the rate of freezing, and the permeability of the surrounding material and the distance it must go to obtain relief. These pressures produce triaxial dilation of the concrete, and if these hydraulic stresses remain below the strength of the concrete, they will rapidly be dissipated, but if they rise above the strength, they will produce permanent damage—frost deterioration. These hydraulic stresses as well as additional later stage strains due to ice accretion or osmotic pressures are often observed in the detailed study of the mechanism of frost action. They can most effectively be eliminated by the presence of air voids of proper number and size.

The relative volumes and the different sizes of pores in cement paste and different aggregates have a large and important effect when freezing occurs. In saturated pastes and aggregates it is only that water that is evaporable at about 80 per cent relative humidity that will freeze at normal winter temperatures. Saturated pastes may have a relatively high capacity for freezable water and still have a very low permeability. With this combination, to avoid development of destructively high hydraulic pressures on freezing requires that an escape boundary for the explusion of excess liquid be very close to the freezing site. In pastes this critical distance is in the range of approximately 0.12 to 0.25 mm (0.005 to 0.01 in.), the escape boundaries being introduced into paste by properly entrained air. This critical distance has been theoretically deduced and also experimentally verified by estimation of the average "spacing factor" of the air-void system in hardened concrete using microscopical techniques (ASTM Recommended Practice C 457). Fine grained aggregates, such as chert, have a lesser capacity for freezable water but still have a relatively low permeability and have critical dimensions on the order of 0.5 in. Above this size they may cause deterioration or popouts in concrete during freezing. Some aggregates have a very coarse pore system and have critical dimensions for unconfined freezing of the order of several feet. Such aggregates may influence the frost resistance of concrete by other mechanisms.

Osmotic Pressure:

It is well known that under certain circumstances concrete prepared with cement of high-alkali content and reactive siliceous aggregates of particular quantity and size distributions can produce an alkali-silicate type of reaction product that may cause expansion of the concrete. Pore pressure can be conceived as the cause of the deleterious expansion, whether the alkali-silicate product is gel-like (semi-rigid, swelling pressures) or fluid-like (osmotic pressures). The fluid-like product to which the paste membrane in the concrete is relatively impermeable tends to imbibe water and dilate, applying destructive pressure to the walls of the pores in which it is confined.

Effective Pore Area:

The magnitude of pore pressure, due to freezing or deleterious reaction, that is required before destructive forces develop in the concrete is related to the effective area of the concrete over which they produce stress. Small and isolated pockets of high pressure might be accommodated by the concrete, whereas lower pore pressures operating over large areas of the concrete could produce forces above the inherent strength of the concrete and could, therefore, cause damage. This problem of the effective pore area in concrete is also of direct concern to the designers of gravity dams. Various mechanical and analytical problems confront investigators of this matter, and unanimous agreement has not been reached concerning the effective pore area to water in mortar or concrete, reported values ranging from about 40 per cent to almost 100 per cent, depending upon the grade of materials studied and the method of analysis applied. That the effective pore area is high is not surprising when the high porosity and fine texture of the pores in concrete are considered.

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Hardened Concrete

CORROSION OF REINFORCING STEEL

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Under most conditions portland cement concrete provides good protection to embedded reinforcing steel against corrosion. The protective effect of the concrete may be ascribed to its high alkalinity, its relatively low permeability, and its relatively high resistance to the flow of electric current.

Notwithstanding the immunity of steel against corrosion that is usually afforded by concrete, cases have been reported in which corrosion of reinforcing steel by galvanic action has necessitated large expenditures for repair and maintenance [*1–8*].[2] The conditions leading to distress have been related to failure of the concrete to provide one or more of the protective factors named in the first paragraph.

Corrosion Cells[3]

Corrosion is accompanied by a flow of electric current within the system and by chemical reactions [*9*]. The flow of electric current may result from an impressed potential from an exterior source which may produce electrolysis [*10*] but, in the absence of an exterior source, may be developed by galvanic action within the system itself.

The electric current emanates from and terminates on two surface locations of metal that are in contact with an electrolyte. The electrolyte may be any substance in solution that is capable of conducting electric current by ionic flow. Portland cement concrete contains such a substance. Its conductivity varies over a wide range, is lower in drier concrete, and becomes higher with increasing moisture content.

The two locations on the surface of the metal that are affected by the flow of electric current are known, respectively, as the anode, where corrosion occurs, and the cathode, where corrosion does not occur. The anode, the cathode, the metal connecting these locations, and the electrolyte constitute the corrosion cell.

Corrosion at a steel anode may be represented by the following equation:

$$\mathrm{Fe} \rightarrow \mathrm{Fe}^{++} + 2e^{-}$$

To maintain equilibrium of the system an equivalent amount of hydrogen is plated out on the cathode. If the film of hydrogen is not removed, further progress of the anodic reaction is inhibited. Under this condition, the cell is said to have become polarized. In order for the reaction to proceed further, the cell must be depolarized by the removal of the hydrogen film in some manner. Depolarization may occur by the evolution of hydrogen as a gas and its subsequent removal from the environment, or by a

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

[3] This discussion is not exhaustive and in some respects is oversimplified. For more complete exposition, consult the texts cited in the list of references.

chemical reaction of the hydrogen with another element. The most common depolarizing element is oxygen in solution, which combines with hydrogen to form water. If the concrete is dry or if air can not penetrate through the surrounding concrete freely, the corrosion cell will remain polarized or its activity will be reduced.

In the absence of cathodic polarization, ferrous ions accumulate at the anode and, in the presence of water and oxygen, are oxidized and precipitated as rust, Fe_2O_3-nH_2O. If the supply of oxygen at the anode is plentiful, the rust may form in contact with the metal and provide a protective film that is capable of inhibiting further corrosion, or reducing the rate of corrosion, independent of reactions at the cathode. However, in the case of concrete, the supply of oxygen is generally limited with the result that the more soluble ferrous compounds are in contact with the metal, and the insoluble rust forms at a point removed from the metal surface and does not provide an effective protective film. In concrete, therefore, stoppage of the flow of electrric current is brought about most commonly by polarization at the cathode. Thus, when corrosion of reinforcement can occur, the condition leading to its inhibition is primarily a very low degree of permeability of the concrete, which prevents much change in the original interior environment.

Electrolysis occurs when a reinforced concrete member forms part of a direct electric current circuit, that is, when an independent conductor is not provided to complete the circuit or when there is leakage from such conductor to the concrete. The mechanism by which corrosion is produced under electrolysis is similar to that in galvanic cells; however, the magnitude of the impressed potential may be many times greater than that generated internally and, hence, may be more destructive.

Point-to-point differences in the metal, such as discontinuities in mill scale, variations in metal structure, or stresses in the metal may be conducive to the production of electrical potential and, hence, give rise to small localized corrosion cells. From the standpoint of widespread corrosion, however, these factors are less important than large scale cells that may be formed. Indeed, reported cases of severe corrosion of normal reinforcement are related predominately to the latter cause. However, pitting, developed in small cells to a degree that may not be of consequence in large reinforcing bars, may be serious in prestressed wires of small diameters [*6*].

Reinforcement in concrete structures generally consists of systems of individual bars or wires that are interconnected electrically by contact, through tie wires or welds. Thus, the entire system may develop large-scale corrosion cells of considerable extent, if conditions are right for their formation and continued operation. Anodic and cathodic areas separated from each other by distances of 2 to 10 ft under potential differences of as much as 0.4 v have been measured [*5*]. Under such conditions serious corrosion leading to cracking of the concrete due to the pressure of accumulated corrosion products has been observed. In advanced cases, spalling of the concrete from the plane of the reinforcement has occurred. If the cathode area is large in relation to the anodic area, localized pitting deep enough to impair the strength of large reinforcing bars results [*5*].

HETEROGENEITY OF THE ELECTROLYTE

The characteristics of the concrete and its electrolyte have been shown to exert a dominant effect on the corrosion process. Aside from its moisture content, affecting conductivity, and its imperviousness, affecting polarization, characteristics of nonuniformity from location-to-location are conducive to the formation of large scale corrosion cells. Such heterogeneity may consist of varying moisture content, varying permeability affecting the availability of oxygen at the steel surface, varying distribution of extraneous salts if they are present, and others [*5,8,11*]. Cells produced by these means are known as differential concentration cells. It is probable that large scale corrosion cells are formed by one or more of such conditions and that they are responsible in the main for severe corrosion, when it occurs, in most concrete structures.

REDUCTION OF CONCRETE ALKALINITY

The pH of concrete normally is higher than 12, depending somewhat on the percentage of alkalies in the cement [*5,12*]. If a highly alkaline condition is maintained at the surface of the steel, there is little probability that iron will go into solution at the anode [*13*]. However, this condition is not always maintained.

Carbon dioxide from the atmosphere may react with calcium hydroxide derived from the cement, forming calcium carbonate, thus lowering the alkalinity. However, carbonation is not presently believed to penetrate deeply into reasonably dense concrete [*14*]. Corrosion from this cause probably occurs only when the reinforcement is but a fraction of an inch from the surface.

Steinour [*15*] states that the calcium hydroxide content of hardened cement paste is probably a fairly good measure of reserve basicity. He notes that in hydrated portland-pozzolan cement the calcium hydroxide content is reduced by the reaction of the pozzolan and that portland blast-furnace slag cement produces less calcium hydroxide than does the equivalent portland because of the dilution. He notes that autoclaved products to which a large proportion of reactive silica has been added may have less than one per cent of calcium hydroxide by weight of the cement.

Monfore and Verbeck [*6*] observed that when corrosion of high-strength steel wires occurred in concrete the corrosion products were located adjacent to a visible void in the concrete that was adjacent to the wire. When fresh concrete bleeds after being placed, there is a tendency to create water voids on the lower surface of the reinforcement. Hausmann [*16*] applied impressed voltage to steel that was thinly coated with hardened cement paste and calculated that a pH value lower than seven was produced at the surface of the metal after the alkaline constituents of the cement paste had been exhausted. Thus, overly dry, overly harsh, or overly wet concrete, when mixed, may produce voids in the hardened concrete in contact with the steel surface and provide a mechanism whereby the pH at that point may be reduced below a safe value. Friedland [*17*] concluded that fresh concrete of plastic consistency provided better protection when hardened than that which is too dry or too wet.

EFFECT OF SALTS

The addition of sea salts to cement paste has been found to lower the pH value. Under high concentrations a value as low as pH 10 has resulted [*5,12*]. Concomitantly the half-cell potential of immersed steel was lowered [*5*]. The concentration of sea salt necessary to effect a substantial lowering of pH is a distinct possibility in concrete in a marine location when the contained moisture has evaporated partially. Normally the anode reaction of steel in concrete produces ferrous hydroxide, but, if chlorides are present, the much more soluble ferrous chloride may be formed. The great majority of reported cases of reinforcement corrosion have been under marine, inland-salt, or applied-salt conditions. Although the usual influence of salts is to enhance the corrosion process, complete or partial inhibition may result in abnormally high concentrations [*8*,*18*], a greater degree of inhibition being obtained with sulfates than with chlorides [*13*,*19*].

Beaton and Stratfull [*7*] examined 187 highway structures in California for evidence of corrosion of the reinforcement in the visible parts of the substructure (piles, columns, and walls) and related it to the concentration of chlorides (Cl) in the immediate environment (soil or water). They observed that approximately 50 per cent of structures had visible evidence of corrosion in the age group of 5 to 25 years when the environmental chloride concentration exceeded 500 ppm and in the age group of 25 to 46 years when the concentration exceeded 100 ppm. Thus, the designed cover over the steel (2 in.), and the general quality of the concrete (6 sacks of cement per cubic yard, probable water-cement ratio from $5\frac{1}{2}$ to 6 gal per sack) did not insure permanent freedom from corrosion under relatively modest concentrations of environmental chlorides. When the environment was free from chlorides only four per cent of the structures had evidence of corrosion. The minimum evidence of corrosion was taken to be the appearance of a crack parallel to the reinforcement. Thus, the minimum criterion, while not implying immediate need of repair, suggests that repairs may be required at some future time. On the other hand, it may be more economical to accept some maintenance expense than to use sufficient cement to produce a completely safe water-cement ratio.

It can be deduced, also, that if the concrete as placed contains chlorides the probable corrosion-free life will be reduced.

Hausmann [*16*] states that "in buried or submerged reinforced concrete structures, corrosion of the reinforcement will be prevented even in a high chloride environment if an alkaline environment is maintained at the steel surface and free oxygen is excluded." Normally it is expected that free oxygen will be excluded or greatly restricted in submerged or buried structures. The maintenance of an alkaline condition at the surface of the steel may not, however, be accomplished in practice.

Griffin and Henry [*18*] cast corrosometer probes in concrete specimens that were sealed above water. The water-cement ratio (W/C) of the concrete was 0.70, and it was mixed with water of varying salinity (sodium chloride or sea water). The probe consisted of wire in a loop about 1 in. in length and about $\frac{1}{4}$ in. in over-all width. They concluded that mixing water not having a salinity in excess of 25 g/kg (1.7 per cent NaCl by weight of cement) did not corrode steel significantly. This limiting salinity is approximately equivalent to a mixture of 73 parts sea water and 27 parts fresh water. From the reported data, it is estimated that such concrete contains approximately 0.20 per cent by weight of salt. Their findings appear to be at variance with the reported long-time experience of the California Division of Highways that salt contents of approximately one half of this amount have resulted in serious corrosion [*5*]. The discrepancy appears to result from a difference in conditions. The restricted volume of concrete surrounding the small probes presumably was sufficiently uniform in composition and salt content that no type of differential concentration cell was formed, whereas such cells predominate in bridge structures in which reinforcement has corroded [*5,8*]. Thus, it appears that many laboratory experiments should be interpreted as to the probability that differential concentration cells were, or were not, developed.

Monfore and Verbeck [*6*] found that serious corrosion occurred in stressed high-strength wires that were embedded in concrete containing from two to four per cent of calcium chloride by weight of the cement, whereas those in concrete without calcium chloride did not corrode under the same test conditions.

There is evidence [*6,20*] that concrete, made with cements that are extremely low in C_3A, releases more free chloride and develops more corrosion under exposure than when the C_3A content is high. However, the reduction in free chloride apparently is not increased significantly by increase of C_3A content above four per cent. Since sulfates are associated frequently with naturally occurring chlorides, the use of a cement that is relatively high in C_3A may lead to sulfate attack and should be avoided for this reason.

Roberts [*20*] found that steam curing of concrete made with a cement containing nine per cent C_3A produced more corrosion than curing at normal temperature, although this finding was not confirmed by Monfore and Verbeck [*6*]. Roberts' results may possibly be explained in part by the finding of Higginson [*21*] that the permeability of concrete is greatly increased by steam curing unless steaming is followed by a substantial period of moist curing at normal temperature.

Monfore and Verbeck [*6*] found that the addition of four per cent calcium chloride by weight of cement produced corrosion that was similar in amount to that produced by an equivalent amount of sodium chloride. They found that a reduction in added calcium chloride from two to one per cent produced somewhat less corrosion. However, they found appreciable amounts of soluble chloride in hardened cement pastes containing as little as 0.25 per cent calcium chloride.

The California Division of Highways has found definite evidence of corrosion on the interior surfaces of pretensioned stranded cable in steam-cured concrete containing chloride equivalent to 0.24 lb of calcium chloride per sack of cement and has noted some evidence of corrosion when the chloride content was as little as the equivalent of 0.05 lb of calcium chloride per sack of cement. However, in the latter case the chloride was largely present as sodium chloride which was introduced purposely as an impurity in the mixing water.[4] These findings tend to substantiate warnings quoted later against the use of chemical admixtures containing chlorides in prestressed concrete.

EFFECT OF SULFIDES

Sulfides may be introduced into concrete by the use of granulated blast furnace slag either as aggregate or a component of portland-blast furnace slag cement. Bogue [*22*] refers to European reports which state that small amounts of sulfide sulfur impart no deleterious effects on steel reinforcement. On the other hand, hydrogen sulfide has been reported to have been a factor in the corrosion of wires in prestressed concrete tanks [*23*].

THICKNESS OF COVER

Other conditions being equal, the greater the thickness of concrete cover over the reinforcement, the greater should be the resistance to penetration to the steel surface of oxygen, carbon dioxide, and environmental salts. Thus, the thicker cover should postpone the initiation of corrosion. Evans [*24*] sug-

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[4] Personal communication.

gests that thicker covers, by virtue of their greater strength, are dislodged less easily from intimate contact with the steel, may produce "rust stifling," and, by such a mechanism, may afford permanent protection.

Kinneman [*25*] states that $\frac{1}{2}$ in. cover of sound, high-grade concrete will protect steel from salt-water corrosion, although he also suggests 2 to $2\frac{1}{2}$ in. cover for unprestressed concrete and $1\frac{1}{2}$ to 2 in. for prestressed concrete. Friedland [*17*] using concrete with aggregate of $\frac{3}{8}$ in. max size, reported an improvement in corrosion resistance when the cover was increased from $\frac{1}{4}$ to $\frac{7}{16}$ in. but that further increases in cover were without effect. Stanton [*26*] reported that precast pipe shells 3 in. thick with approximately 1 in. cover over the reinforcement, exposed in San Francisco Bay for 36 years, did not disclose any evidence of rusting of the reinforcement except at the junction with the cast in place in the superstructure. Thus, there is evidence that thin covers can, under certain circumstances, provide adequate protection against reinforcement corrosion, but it should be borne in mind that the required thickness is related to the quality of the concrete.

CRACKS IN CONCRETE

Cracks leading from the surface of the concrete to the reinforcement may provide access for carbon dioxide and oxygen in air and for environmental salts to the steel surface. A number of investigators have recommended that, in order to safeguard against corrosion of reinforcement in members exposed in aggressive environments, the width of cracks measured at the concrete surface should not exceed about 0.01 in. [*27*]. However, Tremper [*28*] found that cracks of considerable width in blocks exposed for ten years in a moist but substantially salt-free environment did not cause significant corrosion of embedded steel. Hausmann [*16*] states "experience has shown that minor cracks penetrating to the steel are normally not damaging and frequently heal in a moist environment. Protection is also provided at the base of the crack by alkaline material from the adjacent concrete." Gewertz [*4*] discussing the conditions found in the San Mateo-Hayward Bridge in California stated: "The deterioration taking place is found to be independent of surface cracking which normally might be considered to afford easy ingress for the corrosive effects of a marine environment."

As a practical matter, therefore, it does not appear that cracks in concrete are of decisive effect with respect to the development of corrosion.

SUMMARY OF ADVERSE INFLUENCES

With the foregoing in mind, it is apparent that the normal protection of embedded steel in concrete is impaired by:

1. The presence of free moisture in the concrete which increases its electrical conductivity and assists in depolarization by oxygen at the cathode, provided the concrete is not saturated.
2. Perviousness of the concrete which promotes access of salts, if they are present in the environment; access of carbon dioxide; and access of oxygen to the cathodic region.
3. Voids at the steel-concrete interface, which permit a reduction in alkalinity at the anode.
4. Nonuniformity from location-to-location within a member in 1 or 2 above or in thickness of cover.

Conditions that are potentially hazardous are:

(*a*) Reinforced concrete in a wet environment, such that the concrete remains moist but is below saturation.

(*b*) Inadequate provision for drainage

from horizontal surfaces or inadequate conveyance of surface drainage to a point that it can not impinge on vertical surfaces.

(*c*) The incorporation of salts into the concrete as it is mixed.

(*d*) The presence of salts in the environment, for example sea water and its spray, salt-bearing marine sediments particularly if they contain chlorides, salts applied to roadway surfaces for ice control, and brine drippings from refrigerated railway cars.

(*e*) Perviousness in the concrete, brought about by too high a water-cement ratio, poor gradation of aggregates, segregation during placing, or inadequate moist curing.

(*f*) Inadequate consolidation of the concrete or the use of concrete that is too harsh to be consolidated effectively by practical means.

(*g*) Insufficient cover over the reinforcement in relation to the quality of the concrete.

RECOMMENDATIONS AND SPECIFICATIONS

American Concrete Inst. Recommended Practice for Selecting Proportions for Concrete (ACI Standard 613-54) [*29*] recommends that reinforced concrete exposed at the water line, within the range of fluctuating water level, spray in sea water, or in contact with sulfates have a water-cement ratio not in excess of 4.5 gal per bag of cement in thin sections, including piles and pipe, and not in excess of 5 gal per bag in moderate sections, such as retaining walls, piers, girders, and beams. (A footnote provides a permissible increase of 0.5 gal per bag if sulfate resistant cement is used. It appears, however, that such cement, of itself, provides less rather than increased resistance against reinforcement corrosion.)

A report on Durability of Concrete by American Concrete Inst. (ACI) Committee 201 [*30*] recommends a water-cement ratio (applicable to all cements) not in excess of 4.5 gal per bag of cement in all reinforced concrete, except mass sections, that is exposed to sea or brackish water, in contact with more than moderate concentrations of sulfates, chlorides at the water or ground line, or within the range of fluctuating water level or spray. Above this range for a height of 25 ft or within a horizontal distance of 100 ft a water-cement ratio not in excess of 5.5 gal per bag of cement is recommended.

ASTM Standard Specifications for Ready-Mixed Concrete, (C 94) by a note under "Quality of Concrete" recommend that the purchaser in selecting requirements for water-cement ratio with respect to durability be guided by the recommendations of ACI Standard 613-54 [*29*].

An ACI report "Admixtures for Concrete" by Committee 212 [*31*] states:

> Calcium chloride has not been found to promote corrosion of the usual reinforcement in concrete where adequate cover is provided for the steel. However, it should not be used where stray electric currents are expected and should not be used in prestressed concrete because of possible stress corrosion of the prestressing steel.

This report also discusses corrosion inhibiting admixtures and refers to reported beneficial results of sodium benzoate, stannous chloride, and sodium nitrite. Calcium lignosulfonate decreased the rate of corrosion in concrete containing calcium chloride when subjected to an impressed direct current producing an anodic condition in the reinforcement [*32*]. The report concludes with a caution by Evans [*24*] to the effect that, where rust particles occur, there is a risk that the rust, preventing the inhibitor from reaching the metal below, may establish the combination of small anodes and large cathodes—making matters worse.

ASTM Chemical Admixtures for Concrete (C 494 – 63 T) contains the following note:

> Note 2—Admixtures that contain relatively large amounts of chloride may accelerate corrosion of prestressing steel. Where corrosion of such steel is of major concern compliance with the requirements of this specification does not constitute assurance of acceptability of the admixture for use in prestressed concrete.

ACI Standard 604-56, Recommended Practice for Winter Concreting [*33*], contains the following footnote:

> Another exception to the use of calcium chloride may be when there is reason to expect that steel will be corroded by stray currents . . . There are also indications that calcium chloride should not be used in prestressed concrete because of possible stress corrosion of highly stressed special wire steel, but ordinary reinforcement is not affected.

American Railway Engineering Assn. [*34*] states:

> In concrete exposed to sea water the metal reinforcement shall be placed not less than 3 in. from any plane or curved surface and at corners it shall be not less than 4 in. from adjacent surfaces.
>
> In concrete under severe exposure to alkali soils and waters reinforcing steel or other corrodible metal shall be placed not closer than 2 in. from the surface of concrete. In foundations and heavy structures the metal reinforcement shall not be placed closer than 3 in. from the surface.

American Association of State Highway Officials [*35*] requires a minimum cover of 1 in. in bridge slabs without regard to exposure conditions. For conventionally reinforced concrete the following is specified:

> In work exposed to the action of sea water the minimum cover shall be 4 in. except in precast piles where a minimum of 3 in. may be used.

For prestressed concrete the following is provided:

> The following minimum cover shall be provided for prestressing and conventional steel:
>
> | | |
> |---|---|
> | Prestressing steel and main reinforcement | $1\frac{1}{2}$ in. |
> | Slab reinforcement | 1 in. |
> | Stirrups and ties | 1 in. |
>
> In locations where members are exposed to salt water, salt spray, or chemical vapor, additional cover shall be provided.

ACI Committee 201 [*30*] recommends that when salts will be applied to roadway surfaces as an ice control measure the minimum cover over the reinforcement should be 2 in. and that provisions for good drainage of surface water be incorporated in the design.

ACI Committee 201 also recommends a minimum of 14 days of uninterrupted moist curing at normal temperatures and 7 days of such curing following low pressure steam curing.

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Hardened Concrete

CORROSION OF EMBEDDED MATERIALS OTHER THAN REINFORCING STEEL

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This chapter presents a discussion of behavior of metals other than reinforcing steel, plastics, wood, and glass, which are sometimes embedded in concrete for utilitarian or esthetic purposes. Possible detrimental effects on the concrete and embedded material will be considered. These two effects are often, but not always, conjoined.

METALS

Metals other than reinforcing steel which are frequently embedded in or used in intimate contact with concrete are principally aluminum, copper, lead, and zinc. Dry concrete at normal temperature is unlikely to promote corrosion of any of these metals. However, concrete is wet when mixed and for some time after setting, depending principally on the quality of the concrete and the distance through which free water must move to an external surface where it can evaporate. The rate of drying also depends on external humidity, but even at low external humidity a long drying time may be required to lower the internal humidity to a point where corrosion is unlikely. Thus, concrete which will eventually be dry may be internally wet long enough to promote serious corrosion of susceptible metals, and, of course, concrete exposed continuously or frequently to a damp environment may remain wet enough to support such corrosion as could occur. The free moisture in wet concrete provides an aqueous medium which facilitates transport of soluble chemical substances, such as oxygen, calcium hydroxide, alkalies, and chlorides toward the metal, and of any soluble corrosion products away from the metal. It also increases the electrical conductivity of the concrete, thus aiding any tendency for electrochemical corrosion.

The time required for concrete to dry to various internal humidity levels is not commonly appreciated. The rates of drying of 6 × 36 × 36 in. (15 × 90 × 90 cm) slabs of normal weight concrete, as determined by Abrams and Orals [*1*][2] are shown in Table 1.

This table shows that at an environmental relative humidity of 50 per cent, 36 days drying time was required to lower the middepth relative humidity to 90 per cent, and 240 days drying time was required to reach 75 per cent. Additional tests showed that when the middepth relative humidity reached 90 per cent, the relative humidity at a point only $\frac{3}{4}$ in. (1.9 cm) from an exposed face was 87 per cent. The moisture condition of concrete required to support active electrochemical corrosion of aluminum and other susceptible metals is not known with any accuracy, but it seems almost certain that such corrosion could pro-

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[1] Consultant.

[2] The italic numbers in brackets refer to the list of references appended to this paper.

ceed, other conditions being favorable, at 90 per cent relative humidity and probably at 75 per cent relative humidity, though at a lower rate.

Concrete made with lightweight aggregates dries more slowly than that made with normal weight aggregates, and thicker sections, such as beams and columns, will dry more slowly than thin sections, such as walls, or floor slabs not on the ground.

Aluminum:

Aluminum reacts in fresh concrete principally with alkali hydroxides from cement. One reaction product is hydrogen gas, and for this reason aluminum powder is sometimes used in concrete to form cellular or "gas" concrete, or in smaller amounts to provide a slight expansion of grout in bedding machinery base plates. Aluminum in rod, sheet, or pipe form will react much less vigorously than will the powdered metal, because of its lesser exposed area.

TABLE 1—RATES OF DRYING 6 × 36 × 36 IN. (15 × 90 × 90 CM) SLABS OF NORMAL WEIGHT CONCRETE.[a]

Environmental RH	Drying Time to Reach Various Relative Humidities in the Concrete Slab at Middepth, days		
	90% RH	75% RH	50% RH
10	18	80	620
35	30	110	840
50	36	240	. . .
75	36	. . .	. . .

[a] From Abrams and Orals [*1*].

Tests carried out by Jones and Tarleton [*2*] indicate that the corrosion of aluminum embedded in plain concrete can crack the concrete under unfavorable circumstances. However, it has been shown that the situation can be worse if the concrete contains calcium chloride and much worse if it also contains steel, such as reinforcing steel which is connected (coupled) metallically to the aluminum. Wright [*3*] has described a case of corrosion of sufficient severity to cause collapse of aluminum conduit in reinforced concrete containing calcium chloride, and an instance of extensive concrete spalling over aluminum conduit in Washington Stadium has been published [*4*]. Some nine additional cases of concrete cracking over aluminum conduit in reinforced concrete have come to the attention of Portland Cement Assn. (PCA), and in every case calcium chloride was used as an admixture. An example is shown in Fig. 1.

FIG. 1—Concrete spalled by corroding aluminum conduit (from Ref. *5*).

The results of laboratory investigation by PCA have been given by Monfore and Ost [5]. Parts of that investigation will be summarized here, as they bring out the effects of concentration of calcium chloride, alkali content of cement, coupling steel to aluminum, and the ratio of steel area to aluminum area. Six inch (15 cm) concrete cubes were prepared using two cements of alkali contents 0.24 and 0.89 per cent, containing pieces of nominally ½ in. (1.25 cm) aluminum conduit embedded ½ in. from one face. Various numbers of C-shaped sheets of mild steel were also embedded in the cubes, externally connected for some tests and not connected for others.

TABLE 2—CORROSION OF 6063 ALUMINUM CONDUIT EMBEDDED IN 6-IN. CONCRETE CUBES STORED AT 50 PER CENT RELATIVE HUMIDITY FOR 28 DAYS.[a]

Cement	Cement Alkalies as Na_2O, %	$CaCl_2 \cdot 2H_2O$, %	Ratio of Steel Area to Aluminum Area	Electrodes	Days to Cracking	Loss in Surface Thickness, mils[b]
C....	0.24	0	28	coupled	no crack	0.16
		2	14	coupled	5	0.92
		2	28	coupled	4	1.2
		4	7	coupled	3	1.6
		4	14	coupled	3	2.3
		4	28	coupled	3	2.4
		0	28	uncoupled	no crack	0.09
		2	14	uncoupled	no crack	0.07
		2	28	uncoupled	no crack	0.10
		4	7	uncoupled	no crack	0.07
		4	14	uncoupled	no crack	0.07
		4	28	uncoupled	no crack	0.04
		0	0	...	no crack	0.09
		4	0	...	no crack	0.04
D....	0.89	0	28	coupled	no crack	0.12
		1	3.5	coupled	no crack	0.54
		1	7	coupled	no crack	0.77
		1	14	coupled	no crack	1.0
		1	28	coupled	no crack	0.85
		2	3.5	coupled	3	1.0
		2	7	coupled	3	1.4
		2	14	coupled	4	1.4
		2	28	coupled	4	1.6
		4	3.5	coupled	2	1.5
		4	7	coupled	2	1.7
		4	14	coupled	2	2.2
		4	28	coupled	7	3.3
		0	28	uncoupled	no crack	0.33
		2	14	uncoupled	no crack	0.06
		2	28	uncoupled	no crack	0.06
		4	7	uncoupled	no crack	0.07
		4	14	uncoupled	no crack	0.09
		4	28	uncoupled	no crack	0.08
		0	0	...	no crack	0.17
		4	0	...	no crack	0.05

[a] From Ref. *5*.
[b] Calculated from weight losses

Various amounts of calcium chloride were used. The cubes were removed from their molds at 24 hr, coated with a curing compound, and then stored at 73 F (23 C) and 50 per cent relative humidity for 28 days. They were observed regularly for cracks. After 28 days the aluminum pieces were removed, cleaned, and weighed. The principal results are given in Table 2.

Several important findings are to be noted in these results.

1. All cubes that cracked contained calcium chloride.

2. The cubes containing 1 per cent calcium chloride (as $CaCl_2 \cdot 2H_2O$, by weight of cement) did not crack, but metal losses in these cubes were as high as, or higher than, those in other cubes which cracked. One may surmise that exposure to a damper atmosphere might have brought about sufficiently more corrosion to have caused cracking.

TABLE 3—EFFECT OF PROTECTIVE COATINGS ON CORROSION OF ALUMINUM CONDUIT EMBEDDED FOR 28 DAYS IN 6-IN. CONCRETE CUBES CONTAINING 4 PER CENT CALCIUM CHLORIDE AND STEEL COUPLED TO THE ALUMINUM.[a]

Protective Coating		Days to Cracking	Loss in Surface Thickness, mils
Material	Thickness, mils		
None	...	3	2.5
Silicone	...	2	2.1
Lacquer B	1	no crack	0.47
Lacquer C	2	no crack	nil
Bitumen A	5	no crack	nil
Bitumen D	15	no crack	nil

[a] From Ref. 5.
[b] Calculated from weight losses.

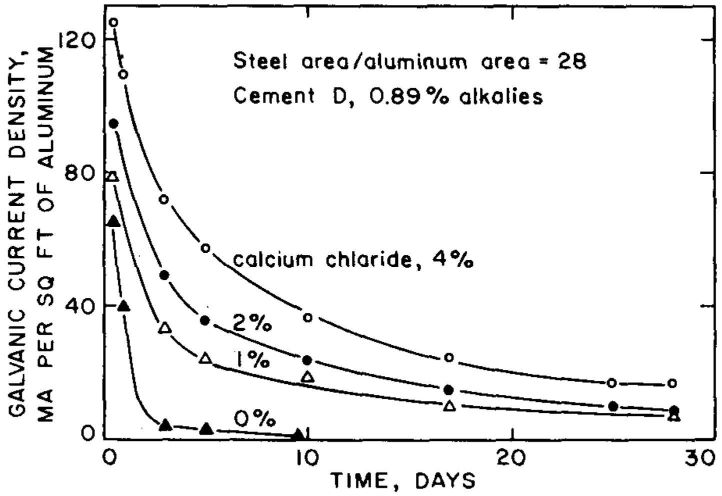

FIG. 2—Effect of calcium chloride on galvanic currents (from Ref. 5).

3. With no calcium chloride and no coupling, the corrosion was greatest with the high alkali cement.

4. With 2 or 4 per cent calcium chloride present, and with metals coupled, corrosion was a little greater with the higher alkali cement.

5. With calcium chloride present, and with metals coupled, corrosion generally increased with increasing ratio of steel area to aluminum area, and invariably increased as the amount of calcium chloride increased.

6. The cubes that cracked did so within seven days.

Additional tests were made to determine the effectiveness of several coatings applied to the aluminum before embedment in 6-in. concrete cubes made with cement of 0.89 per cent alkalies and containing 4 per cent calcium chloride by weight of cement. The results are shown in Table 3. These show that a silicone coating was ineffective and that Lacquer B prevented cracking within 28 days but permitted some corrosion. Lacquer C and bituminous coatings A and D were all effective in preventing both corrosion and cracking.

In the case of coupled metals, considerable galvanic currents were found to flow in the circuit connecting the aluminum and steel. Some measured currents as a function of time and calcium chloride content are shown in Fig. 2. The effect of increasing calcium chloride in increasing the current flow at all periods (up to end of test at 28 days) is evident. When the total electrical flow during 28 days in ampere hours per unit area of aluminum was plotted against the amount of corrosion as loss in thickness, straight lines were obtained with slopes depending on amount of calcium chloride used, the slope being greatest for the higher amounts of calcium chloride. This supports the conclusion that the corrosion observed with calcium chloride and coupled steel and aluminum is essentially galvanic corrosion, the two metals acting as electrodes of an electric cell and the calcium chloride acting as the electrolyte and probably in other ways.

Tests somewhat similar to those reported by Monfore and Ost were carried out on 12-in. (31 cm) concrete cubes by Wright and Jenks [*6*]. With coupled steel and aluminum (area ratio 10:1) the cubes did not crack when no calcium chloride was used, but cracked at various ages from 61 days with 1.1 per cent flake calcium chloride (70 to 80 per cent $CaCl_2$) to 8 days with 5.7 per cent.

The results of these various investigations and field observations show that reinforced concrete is likely to crack and spall from corrosion of uninsulated aluminum embedded therein if an appreciable amount of calcium chloride is present. Whether insulating coatings for aluminum to be used in concrete are commercially practical remains to be demonstrated.

In view of chemical similarities between calcium chloride and sodium chloride, it seems evident that the latter would also facilitate corrosion of aluminum. Sodium chloride is the principal constituent of sea salt, and it therefore seems prudent not to use aluminum in concrete in or near sea water.

Lead:

Lead has a high resistance to certain chemical actions but, in contact with damp concrete, is attacked by the calcium hydroxide in the concrete and becomes converted to lead oxide or to a mixture of lead oxides. If the dampness persists, the attack will continue, and a lead pipe, for example, may be destroyed in a few years. If the lead is coupled to reinforcing steel in the concrete, galvanic cell action may accelerate the attack [*7*], in which case the rate of corrosion may be several millimeters per year. A protective coating or covering should always be used when lead pipe or cable sheaths are to be embedded in concrete. Bituminous coatings have been used successfully. Synthetic plastic coatings or sleeves which are themselves unaffected by damp concrete are suggested.

There appears little, if any, likelihood of concrete itself being damaged by corrosion of lead, because of the softness of the metal.

Copper and Copper Alloys:

In general, copper is not corroded in concrete, although it is reported that thin sheets can become perforated if soluble chlorides are present. Copper pipes are used successfully in concrete except under the unusual circumstance where ammonia is present [*8*]. Very small amounts of ammonia and possibly nitrates can cause stress corrosion cracking. It is reported that brass wall ties have failed by stress corrosion and that manganese bronze bolts have sheared below their ultimate strength, but as such phenomena can occur under circumstances unrelated to concrete, it is not clear what role, if any, the concrete played in these cases. Very little systematic work has been reported on the behavior of copper and its alloys in or in contact with concrete, probably because these metals have given satisfactory service under such conditions.

Rabald [9] says that copper, brass, red brass, bronzes, aluminum-bronze, and copper-silicon alloys have good resistance to corrosion in concrete.

Zinc:

Zinc reacts chemically with alkaline materials, but normally in concrete the reaction is superficial and may be beneficial to bond of zinc coated (galvanized) steel. The primary chemical reaction with calcium hydroxide is

$$Zn + Ca(OH)_2 \rightarrow CaZnO_2 + H_2 \quad \ldots (1)$$

When zinc is used in concrete it is practically always as a coating for steel. The products of reaction are not voluminous and, consequently, damaging stresses are not created.

Although good concrete normally provides a nearly ideal environment for protecting embedded steel from corrosion, galvanized steel reinforcement has occasionally been recommended or used for concrete in a marine environment. Its usefulness has not been adequately demonstrated.

Galvanized corrugated steel sheets are often used as bottom forms for concrete roof or floor construction, the sheets being left in place afterwards. In general, the results have been entirely satisfactory. In some cases they have not. Figure 3 shows one instance of multiple perforation and corrosion of such sheets under a roof slab [10].

FIG. 3—Corrosion and perforation of galvanized steel from under concrete slab (from Ref. *10*)

Most of the corroded spots were dry to the touch, but some were moist. These were found to have pH values of 2.7 to 4.8, definitely in the acid range. Analysis of the corrosion protuberances indicated the presence of iron, zinc, and chloride. Further investigation of this and other similar cases showed that in each instance calcium chloride was used in the concrete and may be presumed to have abetted the corrosion by chemical action on the zinc and by increasing the electrical conductivity of the concrete to corrosion currents. The chemical reaction is probably as follows:

$$Zn + CaCl_2 + 2H_2O \rightarrow CaZnO_2 + 2\,HCl + H_2 \quad \ldots (2)$$

As this reaction produces hydrochloric acid, it may well explain the observed acidity of the corrosion product. It is probable that this reaction could not take place at first but only after local depletion of calcium hydroxide by the reaction expressed in Eq 1.

The extent of corrosion and possible perforation of galvanized sheets used in the manner described with concrete containing calcium chloride is greatly enhanced by use of concrete with high water content and by preventing drying, as by application of a relatively impervious roof deck. Calcium chloride should not be used in concrete to be formed against galvanized steel.

Additional Metals:

Rabald [*9*] reports that the following metals have good resistance to corrosion in concrete: stainless steels, chrome-nickel steels, chromium-aluminum-silicon steels, cast silicon-iron, alloyed cast iron, nickel, chrome-nickel, iron-chrome-nickel alloys, monel metal, stellite, silver, and tin. It is to be noted, however, that the resistance of some of these metals to corrosion in concrete may be seriously affected by the presence of corrosion promoters such as soluble chlorides. Monel metal and stainless steel Type 316 are well known for their resistance to sodium chloride and other constituents of sea water and should work well in concrete. Special circumstances might justify the use of these more costly metals.

PLASTICS

Plastics are used in concrete as piping, shields or sheaths, chairs, and waterstops, and their compatibility with concrete is therefore important. Few, if any, results of tests of plastics in concrete have been published. However, the principal chemicals in concrete which could conceivably attack plastics are calcium hydroxide, sodium hydroxide, and potassium hydroxide. Seymour and Steiner [*11*] list the following plastic groups as having excellent resistance to all three of these alkalies at 75 F:

Polyethylene
Styrene Copolymer Rubber-Resin Blends
Polyvinyl Chlorides, Types I and II
Polytetrafluoroethylene

Another source [*12*] provides the following information on the resistance of plastics to strong alkalies:

Class of Material	Resistance
Polyethylene	excellent
Polymethyl methacrylate	poor
Polypropylene	excellent to good
Polystyrene	excellent
Polystyrene acrylonitrile	good to excellent
Polytetrafluoroethylene	excellent
Polytrifluorochloroethylene	excellent
Polyvinylchloride and polyvinylchloride vinyl acetate (rigid)	excellent
Polyvinylchloride and polyvinylchloride vinyl acetate (plasticized)	fair to good
Saran (monofilament grade)	fair to good
Epoxy (unfilled)	excellent
Melamine (formaldehyde)	poor
Phenol (formaldehyde)	poor
Polyester styrene-alkyd	poor
Urea (formaldehyde)	poor

WOOD

Sawdust, wood pulp, and wood fibers have been incorporated in mortars and concretes, and timbers have been embedded in, or placed in intimate contact with concrete in composite constructions. The use of fresh untreated sawdust, wood chips or fibers in concrete commonly results in very slow setting and abnormally low strength because of interference with normal setting and hardening processes by carbohydrates, tannins, and possibly other substances in the wood. The amount of such substances differs with wood species and from time-to-time and place-of-origin within a single species. Softwoods generally give less trouble in this respect than hardwoods.

Many admixtures and many wood treatments have been proposed or used to circumvent the influence of wood constituents on setting and hardening. Addition of hydrated lime to the mixture in an amount equivalent to one-third to one-half of the cement by volume has been found effective in overcoming this action [*13*]. The treatment is usually effective with mixed softwoods, except when a high proportion of larch or Douglas fir is present. Calcium chloride, to the amount of five per cent of the cement, is sometimes added as well as hydrated lime. With woods of high tannin or carbohydrate content the addition of lime with or without calcium chloride is not effective. Other treatments which have been suggested include soaking in sodium silicate solution, moistening the wood with 1 per cent sulfuric acid for 4 to 14 hr, then neutralizing with milk of lime; treating with 37 per cent aluminum chloride solution or 50 per cent zinc chloride solution in a rotary barrel with beater. A treatment found by Parker [*13*] to be effective with all woods tried consists of the following consecutive steps:

1. Boiling sawdust in water.
2. Draining and washing with water.
3. Reboiling with a solution of ferrous sulfate (2 per cent) in water.
4. Draining and rewashing.

Concrete made with wood aggregate has considerably greater volume change on wetting and drying, or simply on change in external humidity, than concrete made with mineral aggregates. If the element is restrained, drying may lead to cracking. If drying is not uniform, the element may warp. The pretreatments mentioned previously have only a small influence on these volume changes. Various methods have been employed to lessen the changes in volume consequent upon changes in moisture. Some of these methods involve encasement of the wood particles or of the finished product in a material of low permeability to moisture, but the details of such treatments and the results achieved have not in general been revealed.

Timbers embedded in concrete have sometimes been observed to deteriorate. It is said that the harm is done by calcium hydroxide which causes dissolution of lipins and decomposition, chiefly of pentosans, to a smaller extent of lignin, and least of all of cellulose. The most suitable wood for embedment is said to be pine or fir, preferably of a type with high resinous content [*14*].

GLASS

Glass is sometimes embedded in mortar or concrete as artificial aggregate used for decorative or esthetic purposes, as reinforcing as a substitute for steel, as wall blocks or tile, and as frameless windows or lights. In this connection it should be observed that some glasses are expansively reactive with cement alkalies and that the resulting expansion may cause severe damage to the glass or the concrete or both [*20*]. If glasses are to be embedded in or used in direct contact with mortar or concrete, they should be of such character as not to react expansively. The "quick chemical test" (ASTM Test for Potential Reactivity of Aggregates (C 289)), used for the detection of aggregates which are expansively reactive with alkalies, can also be applied to glasses to be used in concrete. That test measures the extent of reaction of the sample during 24 hr immersion in 1N sodium hydroxide at 80 C. Glasses are available which exhibit very low reactivity in the test, both as to reduction in alkalinity and as to silica dissolved. Such glasses are nonexpansive in concrete.

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Hardened Concrete

BOND WITH REINFORCING STEEL

By D. Watstein,[1] Personal Member, ASTM

Synopsis

A review is presented of the significance of various tests of bond between reinforcing steel and concrete. The effect of the more important parameters such as bar diameter, strength of concrete, and spacing of bars, as determined in some recent researches, is appraised, and the appropriate provisions of the standards for reinforced concrete construction are discussed. A brief discussion of the significance of bond and bond tests for prestressed concrete is also included.

Early experimenters and designers of reinforced concrete recognized that slip of the reinforcement had to be prevented in order to develop adequate bond and shear strengths and maintain the integrity of reinforced concrete.

The first comprehensive series of bond tests was conducted by Duff Abrams [*1*][2] who employed both pullouts and beam specimens and measured the slip with care. This investigation laid the foundation for many subsequent studies which finally culminated in the adoption of ASTM Specification for Minimum Requirements for the Deformations of Deformed Steel Bars for Concrete Reinforcement (A 305 – 56 T). This standard defined the minimum requirements for deformed reinforcing bars as we know them today.

Tests for Flexural and Anchorage Bond in Beams

There is a growing recognition that bond stress in a beam may be defined either as the nominal "flexural bond" [*2*] value calculated from the expression $u = V/\Sigma ojd$, or it may be defined as the average bond stress from a point of maximum tension to the end of the bar. The latter has been termed "anchorage" or "development bond," and it is believed to be more meaningful than the "flexural" bond value.

Bond strength is determined either with beam specimens or pullouts. Pullout tests are generally satisfactory for measuring the relative bond values of bars with different deformations. However, experience has shown that bond values derived from pullout tests cannot be applied in general to design of reinforced concrete beams and that beam tests are necessary to develop design criteria.

The differences between bond values obtained from pullouts and beams are due primarily to the different crack patterns which develop in the two types of specimens. Although longitudinal cracks develop along the reinforcing bars in both beams and pullouts, as the stresses increase and slip becomes appreciable, the similarity in the crack

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

patterns ends. In pullout specimens the concrete is in compression, while in the beam specimens both the concrete and the reinforcement are in tension. As the steel stresses increase, transverse cracks develop, and each new transverse crack tends to initiate a new longitudinal crack. Transverse cracks are absent entirely in pullout specimens and the cracking is confined to longitudinal splitting. Mains [*3*] observed in his study of distribution of tensile and bond stresses along reinforcing bars that the tensile stress distribution curves for the beams and pullouts were quite similar when plotted to the same scale and with the free end of the beam bar coincident with the free end of the pullout bar. The similarity of the curves, however, was limited to the portion of the beam between the support and the nearest transverse crack.

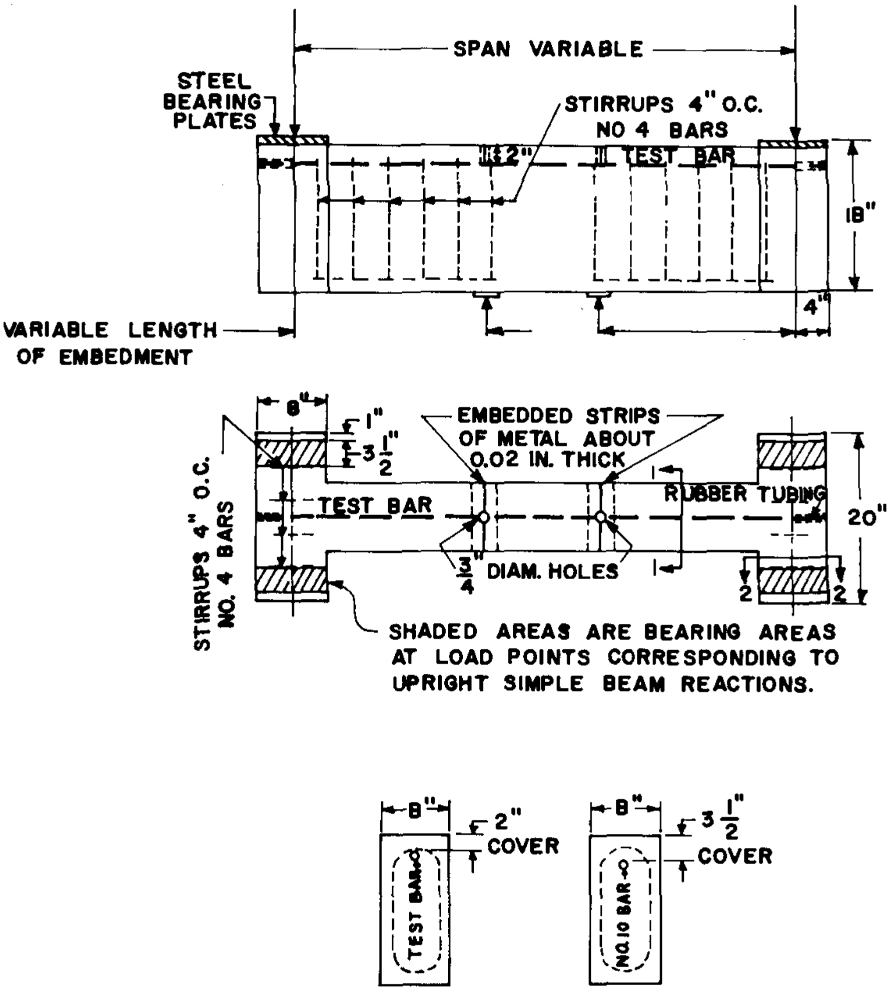
The Bond Stress Committee of the American Concrete Inst. in its report of February, 1945 [*4*] described a test procedure providing a uniform basis for comparison of bond values of different reinforcing bars. This test procedure, which included as parameters length of embedment and position of bar as cast (top or bottom), had been extensively used in bond studies [*5,6*], which culminated in development of ASTM Specification A 305 covering the geometric requirements for deformed bars. The test procedure was later adopted by the American Concrete Inst. as ACI Standard 208-58. This standard did not include any minimum performance criteria since its primary purpose was to establish relative bond values for different bars.

The establishment of minimum requirements for the deformation of deformed reinforcing bars led to a substantial increase in working bond stress for deformed bars conforming to requirements of ASTM Specification A 305. The ACI Building Code for reinforced concrete (ACI Standard 318) adopted in 1951 incorporated new provisions which increased the allowable bond for deformed bars in beams and slabs from 0.05 to 0.10 of the 28-day compressive strength, with a maximum of 350 psi. This code also recognized for the first time the difference between top and bottom bars, as cast. The allowable bond stress for top bars was reduced to 70 per cent of the bond for bottom bars.

Although the test procedure and the test beams described in ACI Standard 208-58 served their purpose well, the standard was rather restrictive since it limited bars to one size, concrete to one strength, and the embedment length to a maximum of 16 in. In order to study the effect of the various parameters on the bond values of deformed bars in simply supported beams subjected to concentrated loads, a beam specimen and a test procedure were developed [*7*], which represent a considerable departure from the ACI Standard 208-58. This procedure, described in the report of ACI Committee 408 [*8*], was intended to provide greater flexibility in the design of the recommended test specimen and test procedure to permit the use of bars of different diameters, more than one strength of concrete, and longer lengths of embedment needed to develop stresses equal to the high-yield strengths of modern deformed bars. This bond test specimen was also intended to provide a research tool yielding data that could be readily translated into design criteria.

Another type of beam specimen designed for investigating bond in the areas of bar cutoffs and points of inflection was used recently in a bond study which included bar sizes from Nos. 3 to 11 [*9*]; two additional bar sizes, Nos. 14S and 18S, were included in a subsequent study by the same author [*10*].

The test specimens described in [*8*]



SECTION 1-1 SECTION 2-2

FIG. 1—Elevation, plan, and cross section of beam specimen, Ref. *8*.

and [*9*] are illustrated in Figs. 1 and 2. The test beam illustrated in Fig. 1 was designed to permit the measurement of the average value of bond stress and the slip at both the loaded and free ends of the portion of the bar between the supports and the load points. The beam was provided with T-shaped ends in order to shift the reactions to points where they would not contribute to the restraint of longitudinal splitting. A strip of metal embedded in the concrete directly opposite each load point assured formation of a crack at that plane, and slip measurements were made at each load point plane.

The test beam shown in Fig. 2 was

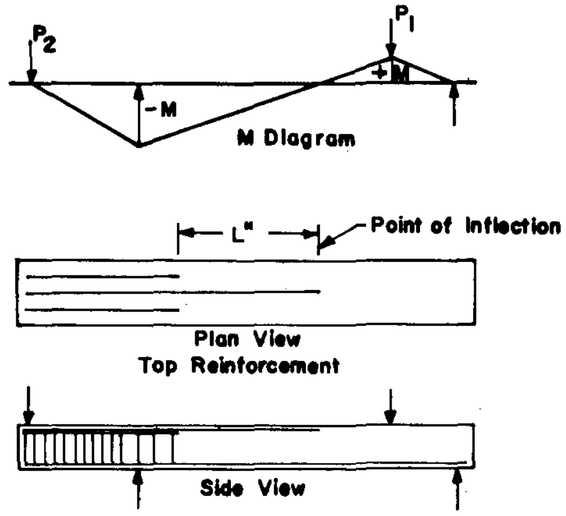


FIG. 2—The University of Texas beams, Ref. *9*.

designed to place the development length of the test bar, designated as L, in a negative moment region between the point of inflection and the point where the other bars were cut off. This arrangement permitted the calculation of the maximum steel stress and the average bond stress in a given development length of the bar. The dimensions of the beam specimen were varied in order to study the effect of the bar cover and beam width.

The two bond investigations described in [*7*] and [*9*] were concerned with different critical sections of beams, and in this sense they may be considered as supplementing each other. The beam shown in Fig. 1 was designed to investigate the bond at the support, while the beam in Fig. 2 was concerned with the bond at the point of inflection and the section where tension bars terminate within a span. In spite of the differences in the test specimens, both investigations have shown conclusively that the bond strength is primarily a function of the bar diameter and that the average bond strength varied about as $\sqrt{f'}$ when other factors were constant. This relationship was recognized by the American Concrete Inst. in the formulation of the allowable bond stresses in the ACI Standard 318-63. This building code for reinforced concrete specifies that the limits for flexural and anchorage bond stresses in bars with sizes and deformations conforming to ASTM Specification A 305 shall not exceed

$$u = \frac{K\sqrt{f_c'}}{D} \dots\dots\dots (1)$$

where:

u = allowable bond stress,
f_c' = 28-day compressive strength of concrete,
D = diameter of bar, and
K = constant which depends on the position of the bar in the beam (top or bottom) and the method of design (working stress or ultimate strength).

For a tension bar conforming to ASTM Specification for Special Large Size Deformed Billet-Steel Bars for Concrete Reinforcement (A 408) (Nos. 14S and 18S bars), the formula for allowable bond stress is given by

$$u = K'\sqrt{f_c'} \dots\dots\dots (2)$$

where K' is defined in Chapters 13 and 18 of Ref. [*2*].

The values of u in Eqs 1 and 2 are limited to certain maximum values irrespective of the bar size and strength of concrete.

Although the bond values for top bars are still limited to 70 per cent of those for bottom bars, there is evidence[3] that for modern low-slump concretes the reduction in bond values for the top bars is not as large as indicated by earlier investigations [*5,6*].

BOND IN LAPPED SPLICES

One of the areas of application in which bond plays a critical role is in tensile lapped splices.

Test data on tensile lapped splices are rather meager [*11,12*], but they indicate that splices in 3000 psi concrete without spiral or equivalent transverse reinforcement are incapable of developing a stress of 40,000 psi with Nos. 6 and 7 bars, or 30,000 psi with bar No. 8 or larger. The 1963 ACI Standard 318 recognized this limitation of lapped splices and recommended that lapped splices be avoided at points of maximum tensile stress and that the splice length be based on a bond stress of only 75 per cent of the usual value.

A recent investigation at the Uni-

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[3] Private communication, R. G. Mathey, Nat. Bureau of Standards, Washington, D. C.

versity of Texas[4] in which test beams of different widths were employed indicated that strengths of adjoining splices decrease as they are spaced more closely. For example, with No. 7 bars lapped 24 diameters the splice developed 39,500 psi for a 6 diameter spacing of splices while for a 3.1 diameter spacing the splice only developed 30,700 psi. This observation is reflected in the new ACI Standard 318 which requires that contact splices, spaced less than 12-bar diameters apart, or closer than 6-bar diameters from an outside edge, must either be enclosed in a suitable spiral or must have a 20 per cent increase in lap.

INFLUENCE OF BOND IN CONTROL OF CRACKING AND DEFLECTIONS

The efficient and economical use of high-yield strength reinforcement is limited by the necessity of avoiding excessively wide cracks which may be objectionable from the standpoint of appearance or lead to corrosion of reinforcement. In recent years considerable research was carried out with new types of high-strength deformed reinforcing bars to study the mechanism of cracking and to determine the effect of such parameters as diameter of bars, ratio of reinforcement, and cross-sectional dimensions of beams on the width and spacing of cracks. Clark [*13*] investigated these variables in a comprehensive series of tests of beams and slabs, and presented a formula relating the average width of cracks to the computed stress in the reinforcement, the diameter of the bars, and the ratio of reinforcement. Chi and Kirstein [*14*] made use of Clark's data in addition to their own to develop a simplified formula for the width of crack, taking into account the distance from the bar surface to the nearest beam surface as a parameter.

The earlier studies by Watstein and Parsons [*15*], and Watstein and Seese [*16*] have demonstrated, by means of tension tests of axially reinforced cylindrical specimens, the direct relationship between the bond strength and both the spacing and the width of tensile cracks. A tension specimen devised to measure the bonding efficiency of a bar [*16*] gave values which correlated well with the spacing and width of cracks observed in an independent set of long axially reinforced tension specimens.

The bonding efficiency of reinforcing bars also affects the rigidity of flexural members because bond stresses affect the contribution of the concrete between tensile cracks to the stiffness of the beam. The resistance of the concrete between tensile cracks to flexure was considered at length in the 1957 *RILEM Symposium on Bond and Crack Formation in Reinforced Concrete* [*17*]. The papers which discuss the various parameters affecting the distribution of strain between cracks and their effect on the flexural stiffness are by Soretz [*18*], Baker, Ashdown, and Wildt [*19*], and Murashev [*20*]. Watstein and Mathey [*21*] discuss the effect of bond on the apparent modulus of elasticity of reinforcing bars embedded in tension specimens simulating portions of beams between tensile cracks.

BOND IN PRESTRESSED CONCRETE

The bond between tensioned tendons and concrete plays a dual role in prestressed concrete beams. One type of bond known as "prestress transfer bond" is developed at the ends of concrete members prestressed by the pretensioning method. In this case, tension in the steel is transferred to the surrounding concrete by bond alone, and the bond stresses serve to anchor the ends of the tendons. The prestress transfer bond is created by the increase in the wire diameter after release of the tensioning

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[4] Private communication, P. M. Ferguson, University of Texas, Austin, Tex.

force, and an increase in wire tension due to flexure reduces the diameter, relieves the radial pressure, and reduces the bond anchorage value near the ends of the beam.

The second type of bond stresses in prestressed concrete is brought about by flexural action. When a prestressed beam is subjected to flexure, the tendons function to some extent as ordinary reinforcing bars and bond stresses develop in accordance with shears and moments in the beam. However, prior to cracking of concrete the stresses in the prestressing steel change quite slowly and the bond stresses are low. As cracking occurs, the steel stresses at cracked sections increase rapidly and bond stresses increase accordingly. These bond stresses which are associated with flexural cracking are commonly referred to as "flexural bond stresses."

In a study of the nature of bond in pretensioned prestressed concrete Janney [*22*] investigated the variation of the prestress transfer bond with various parameters, as well as the interaction between prestress transfer bond and flexural bond. Considerable variation in the anchorage length and the shape of bond stress distribution curves were noted for wires of different diameters and different surface conditions of the wire ranging from rusted to lubricated. Tests of beams prestressed by pretensioning indicated that as cracks occurred the flexural bond stresses increased sharply and the bond stress "wave" progressed from the cracked midsection toward the ends of the beam. As the diameter is reduced and the radial pressure is relieved, the flexural bond anchorage value near the ends of the beam is reduced considerably when the tension in the tendon increases sharply after cracking, and a bond failure may be expected when the flexural bond stress wave reaches the anchorage zone, even before the bond stress concentration reaches the free end of the tendon. Janney [*22*] demonstrated this phenomenon by strain measurements in the posttensioned beams.

As was stated previously, the bond values obtained with pullouts do not always agree with beam specimen data for nonprestressed concrete. This lack of concordance also applies to prestressed concrete. For instance, it will be recognized that in the case of a beam specimen prestressed by pretensioning, the maximum slip occurs at the point of zero stress in the steel, while in a pullout specimen the reverse is true. If a pullout specimen is to be used for the study of bond in prestressed concrete, it should be fabricated in the manner simulating the conditions at the ends of a pretensioned beam.

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ABRASION RESISTANCE

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SYNOPSIS

This paper discusses the various factors that affect the resistance of concrete surfaces to abrasion. The method studied for testing the resistance to abrasion caused by various types of wear indicate that no one test procedure is satisfactory for evaluating all conditions. ASTM Test for Abrasion Resistance of Concrete (C 418 – 64 T) provides a standard test procedure for one type of wear, but there are no standard specifications for use as criteria of wear.

Seldom is there agreement as to what wear actually is or how to measure it. Some feel that the old paint slogan, "Save the surface and you save all," is applicable to concrete. Certainly, when the surface is disrupted and the aggregate exposed, it is difficult to measure the abrasion resistance of the resulting concrete surface. This is particularly true when means are adopted—and some with merit—to increase the life of the wearing surface through the use of concrete surface hardeners or protective coatings.

It has been demonstrated that cement factor, water-cement ratio, air content, and curing each is an important factor. Since all of these factors influence the compressive strength of concrete, it may be reasonably hypothesized that compressive strength may be accepted as a criterion of wear resistance.

It would appear that a reasonable solution to the problem is possible, and an acceptable method of test may be devised, if only concrete and the factors that go into making and curing that concrete are considered. It appears equally obvious that the treatment of concretes to improve the resistance to surface wear involves a different procedure from that used when properties of the concrete mass, as a whole, are the prime factors.

As an example, a very inferior concrete may be painted with a rubber-base paint [*1*],[2] and the resistance to the action of such abrasive influences as sandblasting is extremely high until the painted surface is penetrated. After this surface is penetrated, the resistance to abrasion by comparison becomes ridiculously low. In other words, the strength and toughness of the concrete then become the controlling factors.

The first work which seems to have been reported on wear tests of paving materials was by Johann Bauschinger [*2*] of Munich in 1844. He used a machine, first exhibited at the World's

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

Fair in Paris in 1878, similar to the Dorry machine, in which cores are subjected to abrasion by silica sand carried on a revolving cast steel plate. This work and much of the early work in this country as reported by Page [*3*] and Goldbeck and Jackson [*4*] was conducted on rock and stone then used for paving.

In more recent years, adaptations of these and other methods for determining the abrasion resistance of stone have been used with varying degrees of success on concrete. The investigation of Roman [*5*], Abrams [*6*], Jackson and Pauls [*7*], Scoffield [*8*], and Scholer and Allen [*9*] were with the rattler equipment, such as the Deval test and the Los Angeles and Talbot-Jones rattlers. About the same time, Guttman [*10*] reported on a disk test. A drill press adaptation was also reported by Harris [*11*] in connection with the testing of the hardness of various materials. Tuthill and Blanks [*12*] classified concrete wear into two general types, mechanical and hydraulic. They state that no one test has been devised that adequately measures the wear resistance of concrete under all conditions. This statement still holds.

There are a number of types of abrasion or wear to which concrete may be subjected. Webster's dictionary defines "wear" in this way—"To impair, waste or diminish by continual attrition, scraping, percussion, or the like." The problem seems to be to discover or to select from existing equipment one that could be accepted generally for determining the resistance of concrete to all kinds of wear.

TYPES OF ABRASION

Wear of concrete surface by abrasion will be classified as follows for the purpose of this discussion:

1. Wear on concrete floors, due to foot traffic, light trucking, and skidding, scraping, or sliding of objects on the surface (attrition).
2. Wear on concrete road surfaces due to heavy trucking and automobiles, with and without chains (attrition plus scraping plus percussion).
3. Wear on hydraulic structures such as dams, spillways, bridge abutments, and tunnels due to the action of abrasive materials carried by waters flowing at low velocities (attrition plus scraping).
4. Wear on concrete dams, spillways, tunnels, and other water-carrying systems where a high hydraulic gradient is present. This is generally known as cavitation erosion as distinguished from the abrasive erosion in Type 3 (percussion).

The first type of wear listed is essentially a rubbing action and is usually greatly increased by the introduction of foreign particles, such as sand, metal scraps, or similar materials. Normal wear without the benefit of such abrasive materials would be negligible on a good concrete surface for an indefinite period of time. A concrete which has a high water-cement ratio at or near the surface or has been cured inadequately, of course, would wear down readily.

The second type of wear is caused by a rubbing action similar to that found in the first type, plus an impact-cutting type of wear. This latter type is brought about by the use of chains on automobile and truck tires or metal vehicle wheels. As the wheel revolves, it brings the metal into contact with the concrete surface with considerable impact, a process which tends to cut the surface of the concrete. The presence of wind- or water-borne sand or other abrasive material is a common cause of surface wear on concrete pavements where traffic is heavy. Here again, mix design, finishing, and curing are important factors in the ability of the concrete to resist this type of action.

The third type of wear is primarily a cutting action. The action of the abrasive particles carried by the flowing water, of course, is controlled largely by the velocity of the water, the angle of contact, the abrasive material, and the general surrounding conditions.

The fourth type of wear, cavitation, is completely an impact abrasion [*13*]. It is caused by the abrupt change in direction and velocity of a liquid to such a degree that the pressure at some point is reduced to the vapor pressure of the liquid. The vapor pockets so created, upon entering areas of high pressure, collapse with a great impact, which eventually causes pits or holes in the concrete surface. Also, particles torn loose by this action continue to add to the abrasion problem by causing further wear as designated by the third type previously mentioned. Wallace and Price [*14,15*] have reported on the Bureau of Reclamation's extensive studies of this problem in connection with various dams.

It has been indicated that damage resulting from cavitation is not common in open conduits at water velocities below 40 ft/sec. However, concrete in closed conduits has been pitted by cavitation at velocities as low as 25 ft/sec where the air pressure was reduced by the sweep of the flowing water. At higher velocities, the forces of cavitation are sufficient to erode away large quantities of high-quality concrete in a comparatively short time. This erosion can be minimized, to some extent, by careful attention to form alignment and avoidance of rough uneven surfaces.

The erosion of concrete by silt, sand, gravel, and other solids can be equally as severe as that caused by cavitation. Stilling pools which are not self-cleaning, in which rock and sand collect, are eroded by the movement of the solids by eddy currents in the pool; similarly, concrete over which large quantities of sand and gravel are transported by floods may be eroded seriously.

The concrete in the invert of the 20-ft diameter, 1300-ft-long tunnel at Anderson Ranch Dam was eroded to a depth of about 3 in., while the tunnel was used for diverting the flow of the river for a period of 43 months during construction of the dam. The water carried high percentages of silt, sand, and gravel during the spring runoff, and when the tunnel was unwatered, the invert was covered to a depth of several feet with rocks, gravel, and sand. The wear was fairly uniform on all types of aggregate, and the exposed surfaces of the larger aggregate particles were smooth and flat. Some of the 1:2 dry-packed mortar patches in this tunnel were eroded away completely, and in general the mortar patches were eroded more than the surrounding concrete. The maximum velocity of the water in the tunnel was about 30 ft/sec. New concrete which was installed in this tunnel has been subjected to high velocities of relatively clear water since it was converted to an outlet tunnel. This new low-slump concrete shows only slight wear. Similar erosion was experienced in the diversion tunnels of Hoover Dam prior to their conversion to outlet and spillway tunnels. There are many cases where the concrete of dams and tunnel linings has been damaged by erosion during the construction period, and this possibility should not be overlooked among the design considerations.

Apparently the rate of erosion is dependent on the quantity, size, shape, and hardness of the particles being transported, the velocity of the water, and the quality of the concrete. Concrete-lined irrigation canals which usually carry very few solids show no appreciable erosion after years of service for velocities up to 6 ft/sec.

Where it is expected that the conduit will carry solids or that abrasion will result from solids and eddy currents, the concrete should be of high quality and strength. It is not necessary to be so particular about alignment and surface smoothness where only abrasion is expected and where the velocity of the water will not exceed about 40 ft/sec.

It will be realized, from the foregoing, that probably no one single type of abrasion test can be considered adequate for all conditions. The rubbing type, including rolling balls, may be satisfactory for floor finish surfaces, but in no way does it simulate the cutting action of sand carried by fast-moving water. The dressing-wheel type does approach the cutting action produced by automobile tire chains, but its action is dependent somewhat upon the hardness of the coarse aggregate used in the mix. This is, of course, also true in the case of the rubbing type. The sandblast type cuts into the softer materials, leaving the harder materials exposed or loosened so that they are free to become dislodged from the mass. This type of

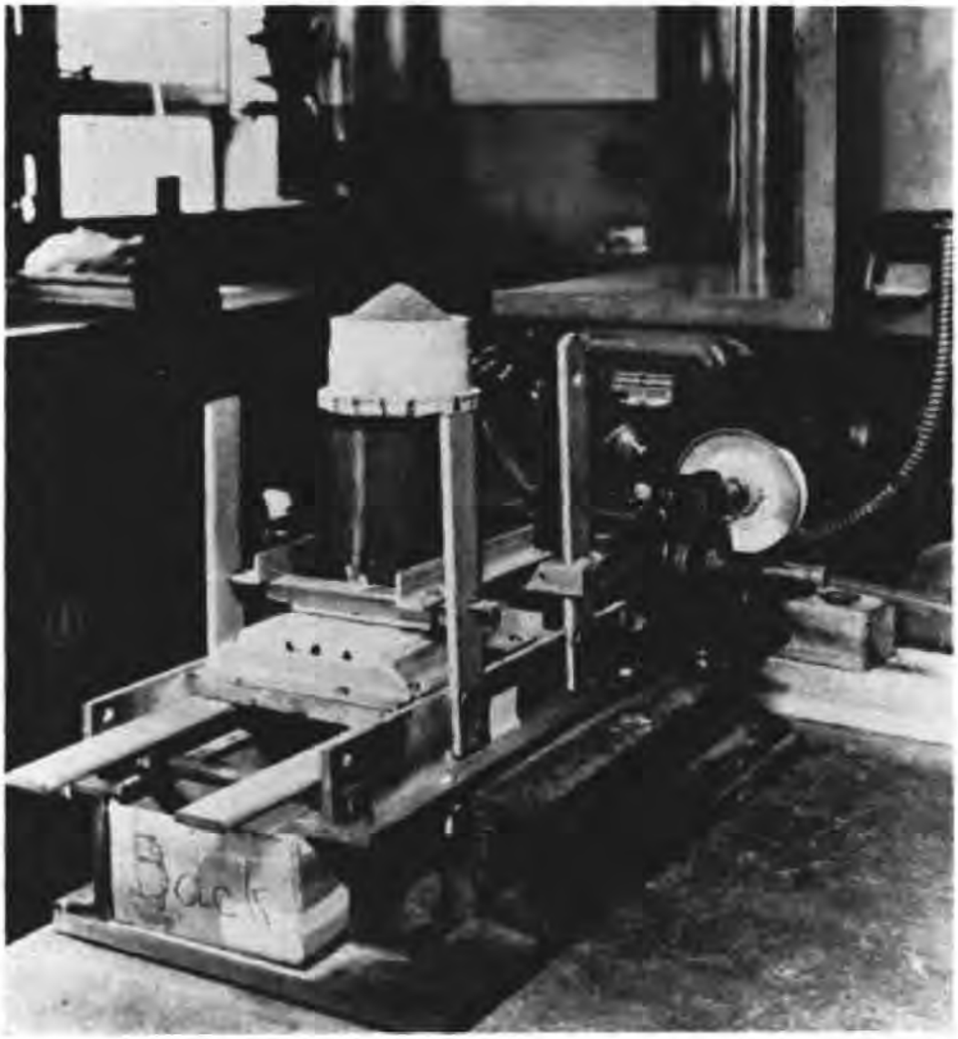
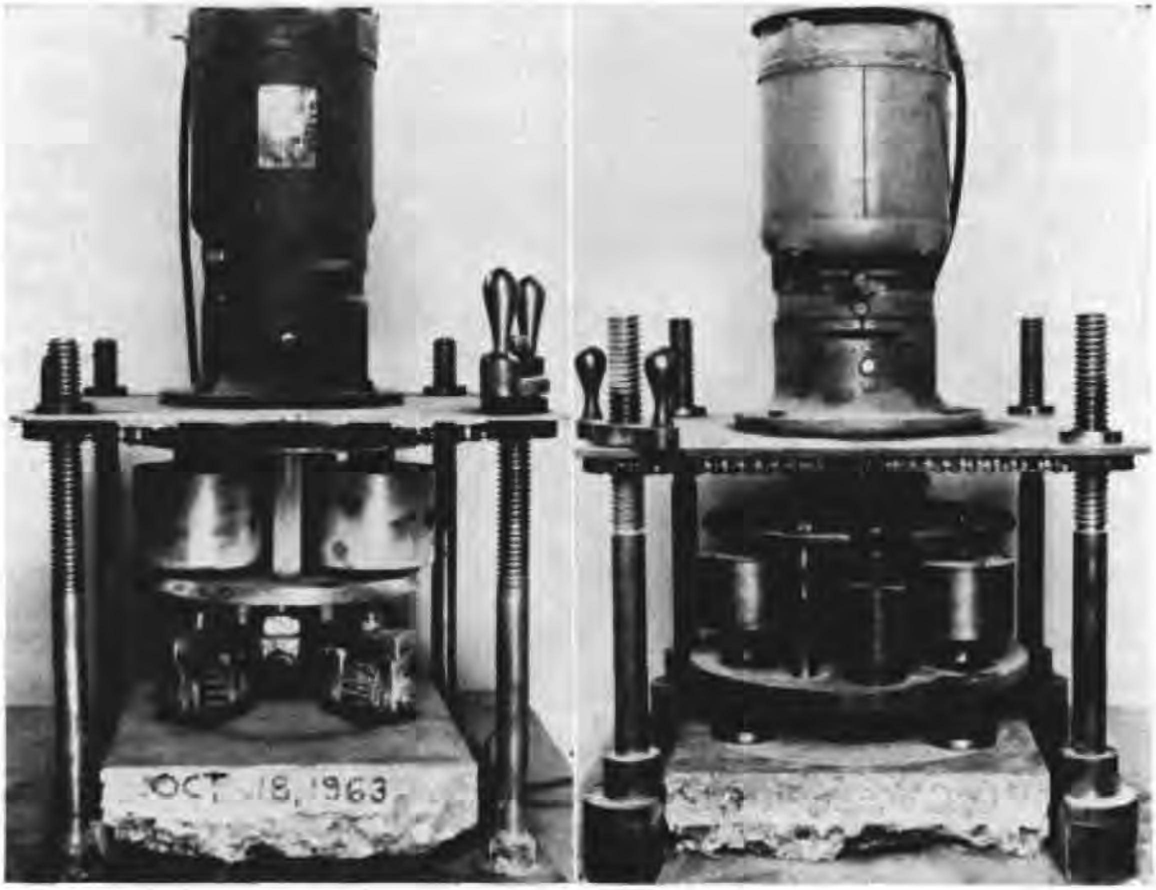


FIG. 1—Reciprocating abrasive machine developed by Research Laboratories, Public Service Gas and Electric Co., N. J.

action is, however, more severe than that commonly found in floors; it cuts through the surface finish and exposes the basic concrete which, under normal conditions of wear, might not be exposed during the life of the building.

All of the methods of test for abrasion resistance that have been investigated by Subcommittee III-m have demonstrated a within laboratory ability to classify concretes of differing abrasion resistance. This fact has been confirmed by Smith [*16*] in his study of the effect of aggregate quality. There are, however, mechanical limitations of the various pieces of equipment which have made it difficult for investigators to recommend a specific piece of equipment. The hardness and condition of dressing wheels at the time of test are examples of the problems encountered. Test data currently available indicate that probably the most reliable and practical method of test is the revolving disk adaptation of the Schuman and Tucker [*17*] machine.



(*a*) Dressing wheels. (*b*) Disks.

FIG. 2—C-9, Subcommittee III-m modification of Schuman-Tucker machine.

TYPES OF SURFACES

Floors:

Wearing qualities of concrete floors have been studied by many investigators. Schuman and Tucker [*17*], Kessler [*18*], Ahlers et al [*19*], Shank [*20*], Emley and Hofer [*21*], Wastlund and Eriksson [*22*], and Sawyer [*23*] have reported on the developments of test methods and results of investigations. This work has involved various finishing techniques, types of curing, time of curing, degree of curing, integral floor hardeners, surface hardeners, and air entrainment. Also included in these studies were the effects of paints, acids, and alkalies on the resultant surfaces with respect to the ability of the surface so treated to resist abrasion.

Most of these studies have been carried out by rubbing types of apparatus [*24*], since this was considered to be the method best suited to reproduce the actual action on the floor surface. Other types—dressing wheel, sand blast, and rolling balls under water—have also been used, but to a limited extent.

The two common methods of achieving this rubbing action are: (1) a reciprocating disk or (2) a revolving disk with some sort of abrasive material in powdered form, such as Carborundum, silica sand, or slag. Some tests have been conducted on specimens under water, but since there is no apparent advantage to this procedure, most of the results have been reported on air-dried specimens. The length of time required to obtain significant results, provided the surface characteristics of the specimen under test are comparable, depends mainly upon the abrasive used, the pressure applied, and the speed of operation. This time may vary from 30 min to 20 hr.

Figure 1 illustrates one of the reciprocating types of machine that have been used in wear test studies.

Figure 2 illustrates the revolving disk type machine currently under investigation by Subcommittee III-m for evaluating floors. It is an adaptation of one developed by Schuman and Tucker [*17*]. This apparatus is portable and can be used on floors in place. This makes it a valuable tool for use outside of the laboratory.

FIG. 3—Drill press operated dressing wheels.

There is also the question of the effect of the aggregate particles, particularly where the surface had no special treatment, such as the use of integral or surface hardeners. Under these conditions, the surface finish wears off quickly, and the subsequent rate of wear will be governed largely by the hardness of the exposed surfaces of aggregate particles. In actual practice, however, the traffic

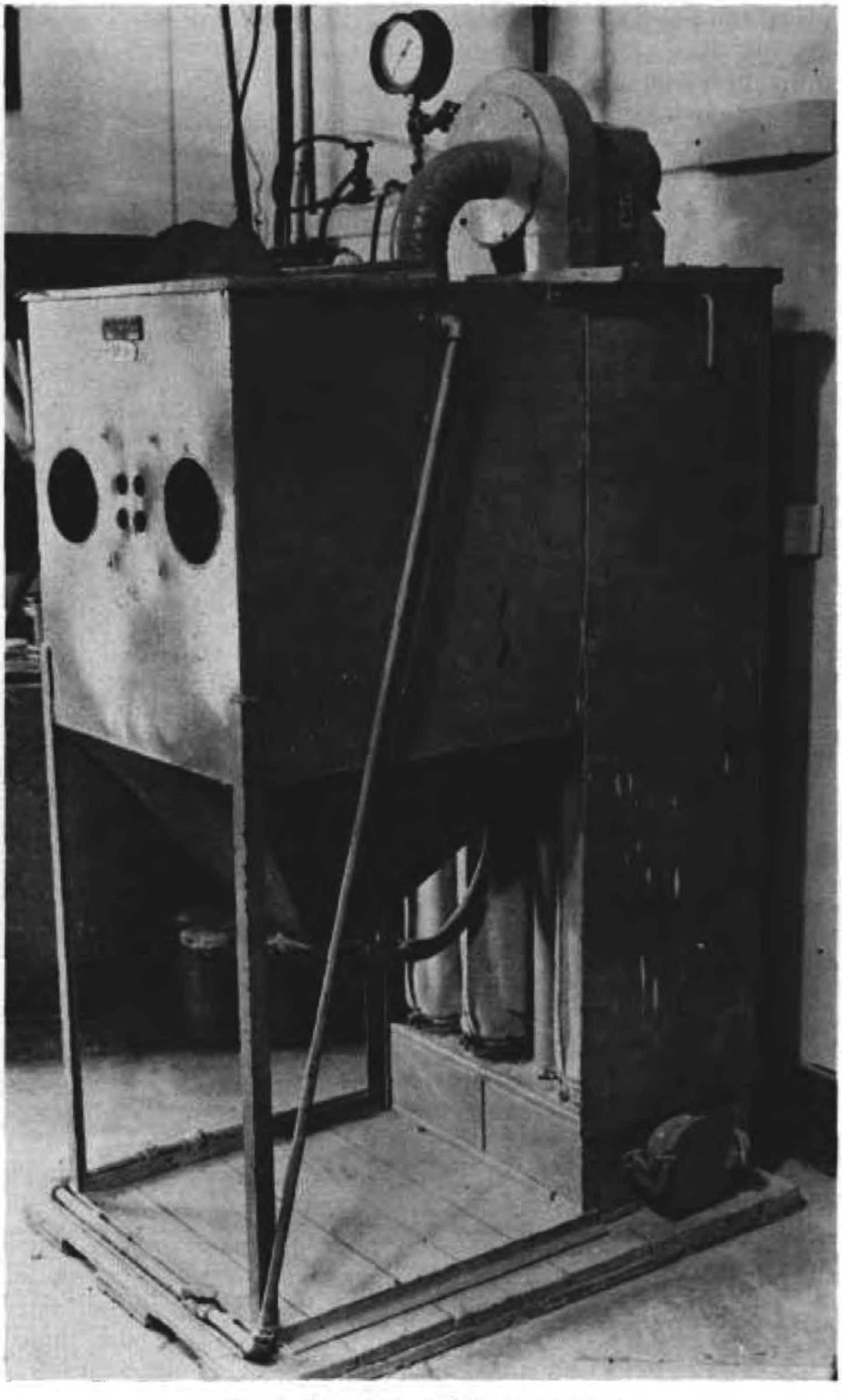


FIG. 4—A typical sandblast test cabinet.

will wear around the harder particles, leaving them protruding and susceptible to impact.

In general, where the conditions of test are such that only a surface hardness is to be examined, the rubbing action will produce satisfactory results. This includes toppings containing an integral hardener, surface hardeners, and various types of finishing, and the application of film-forming materials, such as paints.

The dressing-wheel type of test (Fig. 3) has also been found to be useful for this sort of test. It is a fairly simple piece of equipment, but poor reproducibility of results has hampered its general acceptance. It can be set up in a drill press and does not require an abrasive or water, although abrasives are sometimes used. General practice is to clean the surface occasionally during the test by blowing the dust off of the specimen under test. Some investigation of the use of dressing wheels in place of disks in the Schuman and Tucker machine (Fig. 2) has been carried out by Subcommittee III-m. The program is being continued in an effort to establish a satisfactory test procedure.

When the wear caused by the wheel has progressed through the surface of the concrete, there is again the tendency for the hardest aggregate particle or particles to carry the burden. However, this condition is not so pronounced with the wheel as with the rubbing test, because some of the teeth will be making contact at other points. There will, however, be a tendency for the wheel to bounce because of high and low spots, thus causing gouging. The extent of this bouncing will be controlled to a great degree by the loading pressure applied to the wheel and the speed of operation. The use of an abrasive and lower loading pressures tends to overcome this undesirable action.

It has been found difficult to reproduce the action of the dressing-wheel type of equipment in various laboratories. A breaking-in period seems to be desirable before making an actual test in order to keep laboratory tests within a reasonable range, and a limit on the number of tests per set of wheels is a necessary requirement. Perhaps the greatest reliability or repeatability would be obtained if a set of wheels were used only for a single test. The fact that each unit is made up of several individual cutting wheels, each of which rotates at a different speed dependent upon its distance from the central vertical axis, is conducive to binding between wheels. This results in an erratic dragging and gouging action of some of the wheels and is not conducive to reproducibility. Also, the wheels farthest from the vertical axis travel a longer path and wear down more rapidly.

The sandblast test (Fig. 4) may be successfully used for surface testing, provided due care is exercised to control the rate of abrasion. It is possible to adjust the rate of flow of sand so that within a reasonable period of time it will affect only the surface; thus, the surface treatment can be evaluated. On the other hand, the length of test or the flow of sand can be increased to provide a more severe test that will cut around the aggregate and loosen it, regardless of its relative hardness.

Paints, particularly the rubber-base type, change the effectiveness of the various test methods. In the case of the sandblast there is a tendency for the sand to bounce off the surface without cutting because of the resiliency of the film. Thus, this test becomes of no value as a method of testing the concrete surface. The sandblast will quickly cut through a water-base paint or similar film. However, both the rubbing action and the dressing wheel will wear through the paint film to the surface of the concrete, and it is possible to get a measure of the effectiveness of the paint as a protective coating.

The sandblast test has been set up as a standard ASTM Method of Test for Abrasion Resistance of Concrete (C 418 – 64 T). Originally the test called for either Carborundum or silica sand, but this now has been revised to use only sand. The cutting action of the Carborundum was difficult to control.

Surface-applied floor hardeners also pose a problem for this type of test because they do not generally provide a very thick surface-hardened condition. The milder form of abrasion obtained by the rubbing action or revolving disk is more sensitive to slight differences in surface hardeners than the other types of test methods.

Floors that are subjected to acid conditions, such as in milk plants and citrus fruit packing plants, very often become damaged by the acid action to such an extent that the abrasion resistance is considerably lowered. This reduction in resistance can be measured by all of the methods under discussion. However, in cases of this type, it is the mortar that has weakened; therefore, the sandblast is by far the most effective method. The dressing wheel would rank second, and the rubbing action third. The trouble with the rubbing action, as previously stated, is that it will tend to ride on the aggregate, thus giving a false indication of the true condition of the concrete.

Roads:

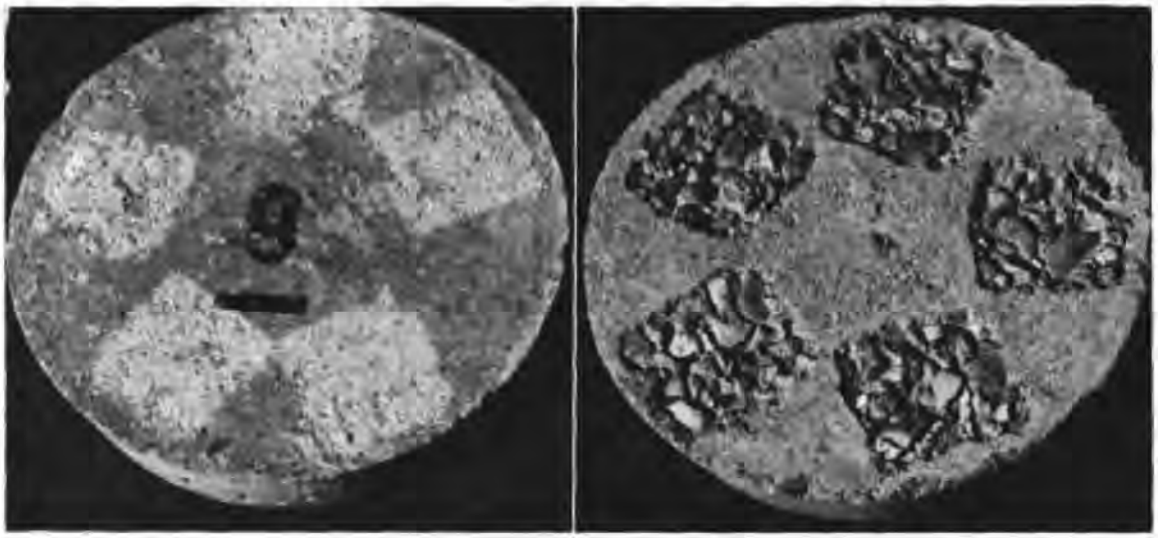
The investigation of the abrasion resistance of roads dates back to the earliest tests that were actually applied to paving stone. Since the introduction of concrete, there have been few published reports concerning the abrasion resistance of concrete roads. Jackson and Pauls [*7*], Collins and Waters [*25*], Wastlund and Eriksson [*26*], and Teller and Davis [*27*] have published reports on abrasion tests of highway concrete. This work is in a somewhat different category from floors, because hardeners, paints, and the like are not used. However, investigations have shown that compressive strength is a very important factor. Data obtained indicate that abrasion resistance increases rapidly with a strength increase up to a certain point, depending upon aggregate, mix conditions, and type of test [*25, 29, 30*], but beyond this point, increases in strength have very little effect on the abrasion resistance. This breaking point in the curve may come anywhere between 4000 and 6000 psi. Collins and Waters' [*25*] data indicate that the initial rate of wear of 2000-psi concrete is about five times that of 4000-psi concrete. They further conclude that the type of aggregate used has an important effect on the later stages of wear of medium- and low-strength concrete. Above 6000 psi the type of aggregate has little effect on the wear resistance. Pogany [*31*] concludes that the abrasion resistance of concrete with hard dense aggregate bears no relation to abrasion resistance of the aggregate alone.

The ability of the road surface to resist abrasion depends primarily upon the mix, placing, finishing, and curing of the concrete. Probably curing is the most important factor of those noted. Improper curing can be very detrimental to the best designed and placed concrete as far as abrasion resistance is concerned. Tests by several investigators have shown the great differences in abrasion resistance caused by various types of curing and times of curing involved. It is well established that the longer concrete is kept moist after the set has taken place the more complete the hydration and the better the strength.

Hence, it follows that, in general, the abrasion resistance of ordinary concrete will be nearly proportional to its actual compressive strength at the exposed surface, a fact that has been borne out by several investigations.

Since the introduction of membrane curing compounds, attention has been directed to the effect of such materials on the surface of the concrete. It has been found that some membrane materials may react with the surface of the concrete in such a way as to inhibit or prevent proper hydration. This action weakens the surface and, consequently, reduces its wear resistance. Under these conditions, it is possible to use any of the three methods of test here described to determine the resistance to abrasion. However, the dressing-wheel and shot-blast methods are to be preferred over the rubbing method because the nature of the road surface is generally such that large aggregate is at or near the surface. Hence, by cutting the mortar, there will be a tendency to loosen the aggregate, thus simulating the action of chains or metal wheels on the road.

Another consideration in connection with membrane curing compounds and their effect on abrasion is the time of application. There is a definite bleeding cycle dependent upon such things as water content of the concrete, water-cement ratio, aggregate grading, cement, temperature of the air, humidity, and wind velocity. Also, the use of purposefully entrained air will bring about a reduction in bleeding. Likewise, water-reducing admixtures which may or may not affect the set of the concrete also have very pronounced effects on the bleeding of the concrete. These factors all have a direct bearing on the ability of the concrete to resist abrasion.



(*a*) Membrane curing compound. (*b*) No curing.

FIG. 5—Two specimens made from the same concrete mix showing the effects of curing on abrasion resistance of concrete.

Tests conducted on specimens that were treated with membrane curing compounds at various periods during the bleeding cycle have indicated that, if they are applied before the concrete has stopped bleeding, the resultant surface has a relatively low abrasion resistance. In other words, the abrasion resistance of the concrete surface increases as the time of applying the curing compound to the concrete approaches the end of the bleeding period. There is little difference in the abrasion resistance if the curing material is applied between the end of the bleeding and the beginning of the drying of the concrete. However, once the concrete has started to lose water by drying after the bleeding has ceased, the abrasion resistance starts to decrease. This, of course, is due to the lack of curing and is somewhat proportional to the degree of curing that is achieved. The extreme is the difference in resistance of cured versus uncured concrete, as shown in Fig. 5. Burnett and Spindler [*28*] have also shown that the time of set, which generally coincides with the cessation of bleeding, is a critical point as far as the relation of application of curing compound to abrasion resistance is concerned. Investigations of the abrasion resistance of concrete, with regard to curing, further illustrate the relationship of strength to abrasion. A poorly cured concrete will have low strength and low resistance to abrasion compared to concrete from the same batch that has been cured properly.

Entrained air influences the resistance of the concrete to abrasion in about the same degree that it affects the strength of the concrete. Generally speaking, concretes containing not over 6 per cent entrained air will not show reduced resistance to abrasion as compared to nonair entrained concrete, provided the mix has been redesigned properly and all other conditions are equivalent [*29*]. It has been noted also that concretes of the same compressive strength, although having a difference of as much as 10 per cent in air content, show approximately the same resistance to abrasion [*30*].

Other factors that tend to affect the abrasion resistance of concrete highway pavements are important but do not usually cause variations in the same project. Variations in the mix, brought about by water-cement ratio, cement content, and aggregate grading, all are reflected in the resistance to abrasion. Generally speaking, these variations follow the changes in strength due to the above factors. The conditions of the base on which the concrete is placed has an effect on the abrasion resistance insofar as the base influences the bleeding. The finishing operation and the resulting fines brought to the surface by this operation will also have a marked effect on the resistance of the surface to abrasion by traffic.

Piles, Footings, Piers, and so on:

As previously stated, the abrasive forces that tend to erode concrete in water are developed by the movement of the water carrying foreign particles. These particles are generally sand and often may actually be softer than the concrete, but the force exerted by the rapidly moving water carrying the sand is such that a cutting action is produced.

The most satisfactory method of duplicating this type of abrasion is obviously the sandblast. However, it is possible to evaluate the concrete to some degree by means of the dressing wheel because of the cutting action.

Concrete for this purpose is useable, even after the skin or surface has been worn off, except where appearance is an important factor. Therefore, in testing this type of concrete, consideration of its ability to resist abrasion is not only given to the surface but also to the basic concrete. After the surface has been cut away, the abrasive forces tend to cut the weaker portion of the concrete, which usually is the mortar, and destroy the bond of the aggregate, thus releasing it from the mass. The sand-

FIG. 6—Davis ball machine.

blast duplicates this action and, therefore, provides an accelerated method of test which cannot be duplicated satisfactorily by other methods. Kennedy [*29*] and Witte and Backstrom [*30*] have reported results by this method.

OTHER TEST METHODS

Many methods of test other than the three mentioned up to this point have been used by investigators with varying results.

One such test involved a modification of the Los Angeles rattler [*8*,*9*]. Concrete cylinders or cubes were placed in the machine and tumbled for various periods of time. The abrasion resistance was then determined by visual observation and by weight loss. This is a rather severe test, involving a pounding action not commonly associated with abrasion. A hard, brittle concrete might break up in this test, whereas a softer but tougher material, might stand up longer.

In actual field practice, however, the harder concrete would resist abrasive forces much better than a relatively softer material. Hence, it is not believed that this method is well suited for the determination of abrasion resistance under such conditions as set forth in this paper.

Another method of determining abrasion resistance is by means of an abrasive wheel. A Carborundum or similar wheel, while moving, is brought into contact with the concrete surface. The specimen may be either wet or dry, and generally provision is made to remove the abraded material during the test interval. This method is of doubtful value because of variations in grinding wheels and variations in individual wheels as they become worn. The wheel will also be supported by the toughest portion of the concrete with which it comes in contact and, therefore, measures the abrasion resistance of the strongest rather than the weakest component.

Other methods involving balls, shoes, and rollers have been used experimentally with varying degrees of success.

One of these, a ball method proposed by R. E. Davis of the University of California (Fig. 6), develops wear by rolling steel grinding media under pressure over the surface of the concrete. Provision is made to avoid tracking of the balls. The surface of the concrete is subjected to flowing water to wash the abraded material off as it is produced. This apparatus is rather bulky and costly, factors which are a disadvantage to the method. Also, tests to date have not indicated that the results obtained are appreciably more consistent, or representative than those of other less expensive pieces of equipment [*26*]. A machine similar to this has been developed in Germany, and test results which have been reported indicate that reproducible results are obtainable [*23*]. This latter machine seems to be very well suited for determining the abrasion resistance of concrete surfaces such as those used on highways, floors, and the like. However, it has one disadvantage in that it is not readily portable and thus is strictly a laboratory piece of equipment.

Significance of Abrasion Tests:

Investigators have used two methods of reporting relative abrasion resistance. One is a record of the weight loss sometimes converted to volume loss, and the other is the depth of wear.

The first method is relatively satisfactory for small laboratory specimens when the weight loss can be determined accurately and is great enough to reflect a significant change. This figure should, however, be converted to volume loss to eliminate possible effects of the density of the material abraded on the reported results. The volume loss should be reported to the nearest 0.01 cm^3 per cm^2 and is the method of reporting required in C 418.

The determination of abrasion resistance by means of a depth gage is applicable to both laboratory and field testing. The procedure followed in this method is to determine the depth of wear for a significant number of points after a definite interval of exposure to the test. The wear should then be reported to the nearest 0.001 in.

Currently, investigations are under way in an effort to develop a method of determining the volume of material abraded by some means other than weight of the sample, which can only be done on laboratory specimens. Such a method would compensate for variations in depth measurements which result from high and low spots that invariably occur as a result of the present testing procedures.

Lack of sufficient significant data from any one test procedure makes it impossible at this time to suggest limits or specifications for the abrasion resistance of any type of concrete surface. Hence, it is necessary to rely on relative values as determined by weight loss, volume loss, depth of wear, or visual inspection for judging the wearing qualities of concrete surfaces.

In order for an abrasion test to be significant, particular attention must be given to the type of concrete to be tested. If it is a regular concrete, without special finishing, then the abrasion resistance can be expected to be a direct function of the concrete strength. If, however, metallic or other hardeners are applied to the floor, such factors must be taken into consideration, and the time required for the abrasion apparatus to penetrate the hardened surface must be determined to give any significance to the test. In this latter case, the abrasion resistance would also correlate with compressive strength, since the strength of surface-hardened material is substantially higher than that of regular concrete. Because of the many factors involving test procedures, the nature of the concrete surface and its intended use, no standard of wear resistance has been established. When satisfactory test methods are available it is probable that such specifications can be developed.

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Hardened Concrete

RESISTANCE TO WEATHERING

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Resistance to weathering and the somewhat synonymous term durability have been used by different authors with meanings varying from very narrow to very broad. In the paper on Resistance to Weathering—General Aspects, published in the 1955 edition of this special technical publication [*1*][2] C. H. Scholer presented a detailed outline of the various factors that may influence concrete durability. This outline subdivides the factors under five major groups as follows:

1. Constituent materials.
2. Construction practices.
3. Physical properties of the hardened concrete.
4. Nature of exposure.
5. Types of loads.

As Scholer pointed out, not all of the factors listed under these headings are of equal importance in their effect upon weathering, and it is still true that many important ones are not as yet well understood, though there has been notable progress in our knowledge in the last decade.

Bauer [*2*] states that durability of concrete is affected by (1) alternate wetting and drying, (2) freezing and thawing, (3) heating and cooling, (4) capillary water, (5) deposition of salts by percolating water, (6) dissolving of certain products (principally calcium hydroxide) by percolating water, (7) the dissolving of cement by certain acids, and (8) chemical reaction between certain constituents of aggregates in high-alkali portland cements. Troxell and Davis [*3*] add mechanical wear or abrasion to this list. Waddell [*4*] begins his list of categories of agencies of destruction with "Deficiencies or weaknesses of the concrete itself, resulting from failure to follow the five fundamentals of good concrete construction." He goes on to point out that these weaknesses, although not actually damaging to the concrete in themselves, may make the concrete more vulnerable to the other three categories which are cyclic forces of the weather, chemical or mechanical attack by outside agencies other than weather cycles, and reaction between the constituents of the concrete itself.

In this paper deterioration of concrete from physical processes produced by exposure to natural weathering will be discussed, such deterioration being of sufficient extent as to impair the usefulness of the concrete in the structure. Other types of deterioration, such as chemical attack and mechanical abrasion and the effects of internal reactions, are considered in other papers of this publication.

FREEZING AND THAWING

Mechanism of Damage:

Damage from freezing and thawing is the most common and the most studied

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

type of physical deterioration from weathering, and a great deal has been learned in the past 25 years about the mechanisms by which damage occurs and the means for preventing it. Much of this knowledge has come from the studies of Powers at the Portland Cement Assn. and others on hardened portland cement paste [*5–12*]. Work is still continuing in this field.

Concrete may be damaged by freezing of water in the paste, in the aggregate, or both, when (1) there is a sufficient volume of freezable water present in the paste or aggregate and (2) when the temperature falls sufficiently low at a sufficiently rapid rate or stays sufficiently low for an appreciable length of time. However, even with both of these conditions present, damage can be prevented, so far as the paste is concerned, by the presence of an adequate system of entrained air voids.

Water in the hardened paste is present in gel pores, in capillary spaces, and, to some extent, in larger voids [5]. The gel pores are too small to permit the freezing of water therein, but water can diffuse from the gel pores to capillaries and larger pores under the influence of various forces. Dilation of the paste structure from freezing can be caused by hydraulic pressure generated when growing ice crystals displace unfrozen water and by growth of bodies of capillary ice produced by migration of water from regions in the surrounding paste to those bodies [*13*]. The rate of cooling is the important variable in damage due to hydraulic pressure, a high rate of freezing causing a greater pressure differential and, therefore, more damage. The growth of ice bodies in large capillaries and on the walls of air voids, however, is promoted by a longer freezing period [*14*].

Damage due to freezing in both pastes and aggregate particles is related to the idea of critical size [*14–18*]. A saturated porous body will not be damaged by freezing if its size is small enough and its distribution of pores and the rate of freezing are such that water expelled by freezing can reach an external surface (or a large pore) and freeze there without exerting damaging pressure on the structure of the body. Aggregate particles not confined in paste are normally potentially less vulnerable to frost damage than hardened cement paste because porosity is usually lower and the pore system is of large dimensions, and the resistance to water flow is therefore less. Also, the strength of rocks tends to be higher than that of paste. When aggregate is embedded in concrete, however, the cement paste forms a membrane around the particles, preventing the escape of water, and particles which are above a critical degree of saturation may dilate and cause distress.

In a hardened paste the relationship to critical size, for a given element of volume, is determined by the maximum distance that water has to travel from any point in the paste to an air void where it can freeze without causing damage. Thus paste, to be protected, needs a system of many bubbles with a small average distance between them. According to Powers [*18*] the average distance between bubbles should be on the order of 50 μ, and if the limit of computed spacing factor, $\bar{L}$, (computed from a linear traverse determination as described in ASTM Tentative Recommended Practice for Microscopical Determination of Air-Void Content, Specific Spacing, and Spacing Factor of the Air-Void System in Hardened Concrete (C 457 – 60 T) less than 0.01 in. is adhered to, this average spacing is probably obtained for the smaller voids, many of which may not be detected in the linear traverse determination. Further work has led to the belief that the computed spacing factor should be smaller than 0.01 in., possibly 0.006 to 0.008 in., depending on the maturity and porosity of the paste. Mielenz et al, in Part 4 of their comprehensive study on air-void systems in concrete [*19*], have found that a satisfactory entrained air-void system in job concrete is assured if ACI 613, "Recommended Practice for Selecting Proportions for Concrete," is followed and an air-entraining agent meeting the requirements of ASTM Specifications for Air-Entraining Admixtures for Concrete (C 260) is used. Under these conditions, computed spacing factors ranged from about 0.004 to 0.008 in.

TABLE 1—FEATURES OF ASTM FREEZING-AND-THAWING METHODS.

Method	Frozen In	Thawed In	Freezing Temperature, F deg	Thawing Temperature, F deg	Freezing Time	Thawing Time
C 290 (rapid water)	water	water	0	40	3 hr max[a]	½ hr min
C 291 (rapid air)	air	water	0	40	3 hr max	1 hr max
C 292 (slow water)	water or brine	water or brine	0	73.4	18 to 24 hr	18 to 24 hr
C 310 (slow air)	air	water	0	40	16 to 20 hr total; 5 to 7 hr to cool to 0 F	5 to 7 hr total; 1 to 1½ hr to raise to 40 F

[a] Entire cycle takes 2 to 4 hr with not less than 25 per cent of time for thawing.

It has previously been thought that, if concrete contained a well-distributed system of the smallest air voids, as indicated by a sufficiently small spacing factor, the total amount of air was of minor importance. However, in view of more recent ideas mentioned above about the growth of ice bodies in larger pores and spaces, it appears that a distribution of larger voids is needed in addition to the required spacing factor and, therefore, a minimum total air content is needed as well. What this minimum figure is has not been established but probably varies with different concretes and may be of the order of 4 to 5 per cent or greater by volume of the concrete.

Further work by Helmuth [*20*] has led to the hypothesis that the mechanism that produces expansion in moist hardened pastes is not the development of pressure in the water in all the pores but the production of localized osmotic pressure at the sites of ice formation. Also in a more extensive discussion of the conditions that must be met for ice formation in capillaries of various sizes, Helmuth [*20*] presents evidence that ice formation requires seeding in supercooled water and then is propagated dendritically through the interconnected pores of the paste. Growth of this dendritic network and resulting expansions during cooling depend primarily on freezing in smaller capillaries at lower temperatures.

Freezing-and-Thawing Tests:

For many years, freezing-and-thawing tests have been used by laboratories in efforts to evaluate durability of concretes or aggregates, or the effect on durability of various materials, treatments, curing conditions, and so on. Until comparatively recently, different laboratories developed their own procedures, which conformed with their own purposes, ideas, and availability of funds for construction of equipment.

In 1952 and 1953, four tentative standard methods of test developed by Committee C-9 on Concrete and Concrete Aggregates were adopted by ASTM [*21*]. These methods were largely representative of the procedures then in use in the laboratories represented by membership on Subcommittee III-o on Methods of Testing Concrete for Resistance to Weathering. The methods are ASTM Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290); Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291); Tentative Method of Test for Resistance of Concrete to Slow Freezing and Thawing in Water or Brine (C 292); and Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310).[3] The chief characteristics of these four methods are given in Table 1. Deterioration of specimens is determined by the resonant frequency method, ASTM Test for Fundamental Transverse Longitudinal, and Torsional Frequencies of Concrete Specimens (C 215). Descriptions of apparatus which have been used to conduct tests using some of these methods are given in Refs. [*22–27*].[4]

Two studies reported since these methods were adopted attempted to evaluate the adequacy of the methods for discriminating between concretes of differing durabilities in the same or in different laboratories. The first of these was a study in one laboratory, the National Bureau of Standards, which used all four methods in testing a variety of concretes [*25*]. The other study was a cooperative testing program conducted by Highway Research Board (HRB) Committee MC-B1 on Durability of Concrete—Physical Aspects, in which 13 laboratories participated using three concrete formulations [*28*]. Some of the general conclusions from these two programs were as follows:

1. Methods involving freezing in water were more severe, that is, produced failure in fewer cycles, than were those involving freezing in air.
2. Rapid freezing was more severe than slow freezing when done in air but not when done in water.
3. Rapid freezing and thawing in water (ASTM Method C 290) appeared to do the best job of detecting a difference between concretes both of which had high durability.
4. Only the slow freezing in air and thawing in water method (ASTM Method C 310) was able to discriminate adequately between concretes of low durability.
5. In general, the four methods tended to rate different concretes in the same order of durability when there was a significant difference.

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[3] The two slow methods (C 292 and C 310) have been recently dropped for the following reasons. Neither of the two methods is in general use nor is required by other ASTM specifications which call for freezing and thawing tests. Although equipment for the slow methods is less expensive than that required to run the rapid tests, this advantage is offset by the increased labor and other costs necessitated by the larger number of cycles and longer time required for results. Laboratories that desire to use slow methods for research purposes may still do so and would probably modify the procedure anyway. It has, therefore, appeared desirable to reduce the number of methods specified from four to two.

[4] References [*22–24*] were published before the tentative methods of test but describe apparatus on which some of these methods were based.

HRB Special Report 47 [*28*] drew the following conclusions with regard to the advantages and disadvantages of the four methods:

(*a*) The rapid-water method has the advantage of great severity and a uniform degree of saturation during exposure. It distinguished quickly and decisively between very good concrete and concrete which was less durable either because of insufficient air or because of poor coarse aggregate. On the other hand, the severity of this method was apparently so great that no differentiation was provided between the two less durable concretes. Data from other methods suggested that in many cases differences did exist between these two concretes, as fabricated in the different laboratories, and also within the single class of low-air concrete with good aggregate.

(*b*) The rapid-air method appeared to be less consistent in its distinction among the different concretes, although it showed the best concrete to be uniformly highly durable and the concrete with poor aggregate to be nondurable. Wide differences existed among laboratories for the low-air concrete with good coarse aggregate. Study of the data revealed that, although this may have been partly due to between-laboratory differences in the changes in saturation caused by air freezing, the variations in durability factor for the low-air concrete were related primarily to differences in unit weight of the specimens fabricated in the different laboratories. Thus, it appears that this method was sufficiently sensitive to detect the effects of very small differences in air content of the low-air specimens when these allowed the air content to increase to the level at which frost resistance was produced.

(*c*) The results obtained by the slow-water method did not appear to differ significantly from those of the rapid-water method, but the slow-water method required a much longer period to produce the same results.

(*d*) The slow-air method was less severe than the rapid-air method, and it failed to detect the detrimental effects on durability of inadequate entrained air.

One of the difficulties with the four methods which was pointed up especially by the HRB cooperative program was poor repeatability and reproducibility of results within and between laboratories. Good reproducibility is generally obtained only for concretes which are very high or very low in durability. For concretes in the middle range, a wide spread in results is obtained. Attempts have been made to write precision statements for the four freezing-and-thawing methods according to ASTM Recommended Practice for Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material (E 177) using data from the HRB program. This attempt was unsuccessful even when the spread of durability was reduced by selection of data from concretes within a narrower range of air content than was allowed in the program. It is possible that if data were obtained with more than the six to nine specimens per batch recommended in HRB Special Report 47 and with a smaller range of air contents than was allowed in that program, this problem could be attacked successfully.

Some laboratories have experienced difficulty with damage from bulges in specimen cans when using ASTM Method C 290. This difficulty has been manifest, however, chiefly in apparatus in which air rather than a liquid, such as alcohol or brine, was used as a freezing medium. Although this phenomenon has not been thoroughly investigated, it appears that with the slower freezing of the water in the specimen cans that occurs when air is used, the water freezes first at the top of the cans, trapping unfrozen water beneath the ice. Subsequent freezing of the trapped water then bulges the sides of the cans, and in some cases there is even evidence that the freezing water has stressed the concrete specimens in tension beyond the breaking point. Attempts to obviate this difficulty by the use of rubber windows or joints on the cans or by the use of rubber containers have been unsuccessful. The latter method has resulted in a greatly increased length of time for freezing and thawing due to the poorer heat-transfer qualities of the rubber containers used. Thus, it appears that the use of a liquid freezing medium for ASTM Method C 290 is much to be preferred [*25*,*27*].

Powers' Proposed Test:

One of the most common criticisms of the freezing-and-thawing test methods in use is that they do not reflect the conditions under which concrete is subjected to freezing and thawing under natural weathering. Attempts to correlate laboratory tests and natural exposures have met with limited success [*29*,*30*].

The chief aspects in which the test methods differ from natural freezing and thawing are in the increased rates of freezing and thawing and in the increased degree of saturation of the concrete. Powers, who has discussed this problem extensively [*13*], points out that concrete in the field seldom cools faster than 5 F/hr while laboratory freezing rates are often much faster. Also, most concrete in the field, with the exception of that placed directly in a marine or fresh water environment, is usually subject to periods of drying before freezing.

Powers [*13*] has proposed a modified test procedure which recognizes and tries to overcome the above difficulties. The essential features of the proposed method are as follows:

1. Specimens are cured under standard moist conditions for two weeks, stored in laboratory air for two weeks, then stored in water for two more weeks, with observation and recording of length changes.
2. They are then cooled at a rate of 3 C/hr to −18 C, holding the low temperature overnight. They are not immersed during freezing but are prevented from drying.
3. If the specimens dilate on freezing, they are considered to have failed, since two weeks of soaking has rendered them vulnerable to frost damage.
4. If they do not dilate, they are returned to soaking for two more weeks and the freezing repeated.
5. This is continued until the longest safe period of soaking has been found.
6. The specimens may then be subjected to several successive cycles of freezing and thawing in water to establish the characteristic rate of dilation under laboratory conditions.

Powers also discusses whether or not, in the absence of dilation, absorption rate is increased significantly by freezing and thawing and whether the end point should be the first occurrence of *permanent* dilation after thawing or occurrence of dilation (rather than contraction) during the freezing.

The Powers' method has been tried in two published cases with promising results. Tremper and Spellman [*32*] used the procedure to test aggregates in air-entrained concrete for use in highway work subject to severe winter conditions at high elevation in California. Many of the aggregates tested would not have been considered acceptable under commonly used test methods; yet, the concretes containing the aggregates are reported to be giving satisfactory service with no signs of distress after exposure for several winters.

Tremper and Spellman found that no

permanent increase in length occurred unless dilation during freezing exceeded 50 millionths and adopted this figure as the criterion of unsatisfactory dilation in their work. However, attempts to relate permanent change in length to dilations indicated by the strain-versus-temperature curve in such a way as to preclude the necessity of making the latter measurement were unsuccessful. They also found evidence that even air-entrained concrete is not likely to remain permanently resistant to freezing, if exposed in such a way as to be continuously immersed in water, but that even short periods of exposure to drying provided substantial relief to the buildup of vulnerability resulting from long-time soaking.

Wills et al at the University of Maryland [*33*] used a procedure based on Powers' suggestions in a laboratory study of concretes made with different aggregates and compared the results with those obtained by ASTM Method C 291. They obtained a good correlation between the two methods of test and found that in the Powers' method the measurement of residual length change alone afforded as significant a correlation with the rapid freezing test as did the measurement of dilation during freezing.

Powers' concept of the test he proposed is that its nature and purpose are fundamentally different from those of the orthodox tests and that comparisons between tests based on his method and those conducted by the published methods invite an improper evaluation of the usefulness of the former. He has suggested that his proposed test be called "A Test for the Length of the Period of Immunity to Damage by Freezing." In commenting on this section of this paper, Powers has stated:

> My unorthodox belief is that durability is not a measureable property; rate of disintegration in the laboratory cannot have a consistent relation to rate of disintegration in the field; one *can* tell by test whether or not freezing produces internal stress even when the stress originates in just a few pebbles; design and control of frostproof concrete should be based on comprehensive studies of moisture in concrete in relation to environmental factors, not on the results of freezing and thawing tests, at least not as those tests are now interpreted.

All I can add to this is that Powers' beliefs may be agreed with to a greater extent than his claim of unorthodoxy would lead one to suspect.

Assessment of Freezing and Thawing Damage:

From the beginning of attempts to measure resistance of concrete to freezing-and-thawing damage, both in field and laboratory, one of the problems has been how to determine the extent of damage or the incidence of something defined as failure, and particularly, how to do so in some quantitative manner. In the beginning, visual inspection was the only method of evaluation available. Later, quantitative measures were used such as: (1) weight loss, (2) length changes, (3) making flexure or compression tests after a certain number of freezing-and-thawing cycles and comparing the results to those obtained from unfrozen companion specimens maintained under standard curing conditions, and (4) dynamic modulus of elasticity based on resonant frequency measurements in accordance with ASTM Method C 215. In some cases combinations of these methods have been used. For instance in a cooperative program conducted in 1944 by Highway Research Board Committee MC-B1 on Durability of Concrete—Physical Aspects, weight and dynamic modulus were determined. When the reduction in dynamic modulus reached 30 per cent, beams were broken in flexure and compared to companion unfrozen specimens, and modified cube compression tests were made on halves of the broken beams [*34*]. In the later cooperative program sponsored by Committee MC-B1 and reported in HRB Special Report 47 [*28*], weight and dynamic modulus measurements were made and some of the participating laboratories also made length measurements.

The four ASTM Methods (C 290, C 291, C 292, and C 310) all call for measurements of weight and fundamental transverse frequency. Numerical values of relative dynamic modulus of elasticity are calculated by the formula:

$$P_c = \frac{n_1^2}{n^2} \times 100$$

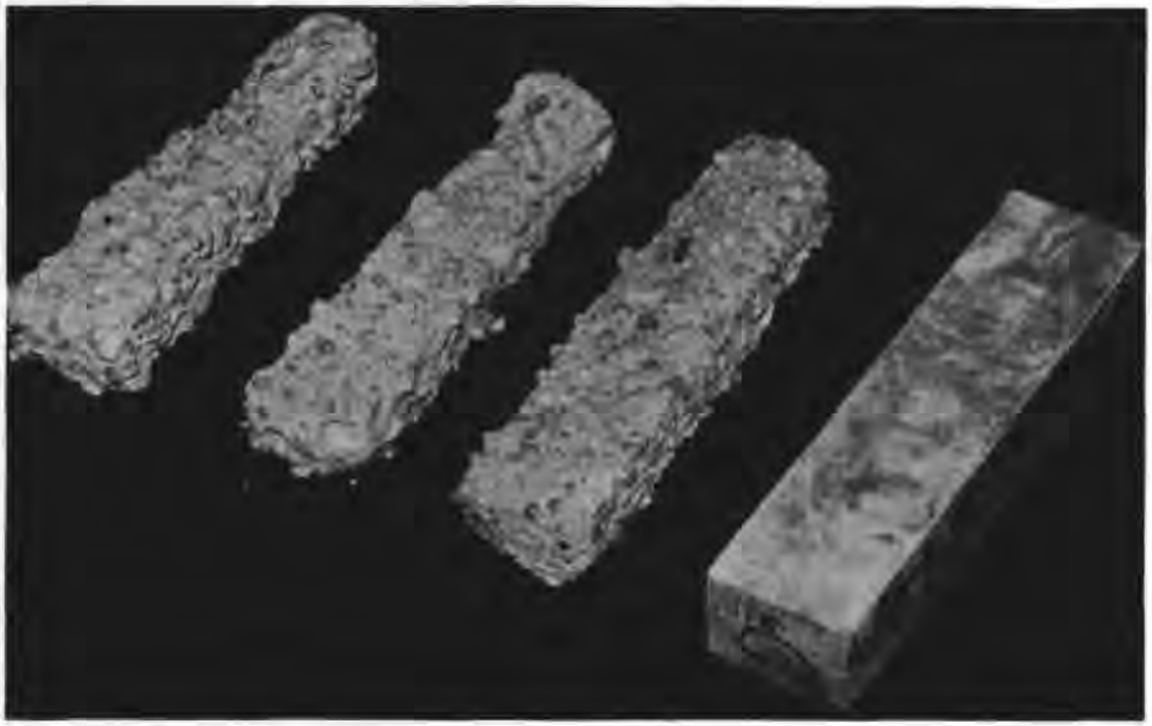

FIG. 1—Surface sloughing of durable concrete specimens frozen and thawed in water according to ASTM Method C 290. Three specimens on left were removed after 300 cycles of freezing and thawing. Specimen on right unfrozen.

The question of which measure of deterioration is best to use is complicated by the facts that the different available tests measure different things, and the manner and extent to which the properties measured are related to freezing-and-thawing damage, especially under natural weathering conditions, are matters of disagreement. Thus, the particular test method used often depends on the philosophy of the laboratory using it and on the particular purpose for which the tests are being made.

where:

P_c = relative dynamic modulus of elasticity, per cent after c cycles of freezing and thawing,

n = fundamental transverse frequency at 0 cycles of freezing and thawing, and

n_1 = fundamental transverse frequency after c cycles of freezing and thawing.

The use of the ratio of the squares of the frequencies in this formula ignores the effects of changes of weight and dimensions of the specimens on dynamic modulus of elasticity, but it is assumed that this method of evaluation is adequate for making comparisons between the relative dynamic moduli of different specimens.

Specimens subjected to laboratory freezing-and-thawing tests may be damaged in one or both of two ways. If the paste is not sufficiently protected by entrained air or a large portion of the aggregate is vulnerable to freezing and thawing damage, the specimens may show a large drop in resonant frequency in a very few cycles, indicating that numerous internal fractures have destroyed the structural integrity of the concrete. If the paste is of good quality, that is, well cured with low water-cement ratio and low porosity, and protected by an adequate amount of air, well distributed in small bubbles, and if the aggregate is sound, internal structural damage does not occur. In this case the specimens may last a long time with very small decrease in P_c, but with successive cycles of freezing and thawing more of the surface sloughs off until the original shape and dimensions are completely lost (Fig. 1). This behavior is due to progressive saturation of the outer layers of the concrete specimen resulting in disruption of the outer layer during the next freezing part of the cycle and is explained quite graphically by Powers [*35*].

The relative dynamic modulus, P_c, as defined in the four ASTM freezing-and-thawing methods ignores deterioration of the latter type, although in many cases such drastic spalling of a concrete surface would constitute failure, since the concrete could no longer adequately serve the purpose for which it was intended. For this reason the following formula involving both weight and resonant frequency has been used [*25*]:

$$P_c = \frac{W_1 n_1^2}{W n^2} \times 100$$

where:

P_c = index of deterioration, per cent, after c cycles of freezing and thawing,

W and n = weight and fundamental transverse frequency, respectively, at 0 cycles of freezing and thawing, and

W_1 and n_1 = weight and fundamental transverse frequency, respectively, at c cycles of freezing and thawing.

With this formula, obviously, P_c decreases with either weight loss or reduction in resonant frequency, and specimens which slough drastically without losing much in resonant frequency are removed from the test earlier than they would be with the use of the ratio of squares of resonant frequencies alone.

The sloughing of specimens in water freezing requires that specimen cans be filled with increasing amounts of water as tests progress. The earlier removal of sloughed specimens is, therefore, desirable for two reasons besides actual recognition of the loss of material as a type of deterioration: (1) the necessity for freezing an increased amount of water changes the conditions of test and (2) the danger of damage to specimen containers is increased by the freezing of the greater volume of water, which allows the water to freeze initially at the top as described above.

Some work has also been done on determining how the constant C, used in calculating dynamic E according to ASTM Method C 215, changes as the dimensions of originally prismatic specimens change due to sloughed off material. Some such correction should be made if an actual dynamic modulus is desired, rather than a relative dynamic modulus or an index of deterioration.

The use of weight loss alone as a

measure of deterioration is open to the same objections as the use of n_1^2/n^2 alone for reasons given above. However, whereas the sloughing without reduction in resonant frequency is a problem mainly of the methods using freezing in water, loss in dynamic E without appreciable weight loss may occur with use of any of the four methods.

The use of length change as a measure of deterioration is based on the fact that internal damage is accompanied by either expansion rather than contraction during cooling or by a permanent dilation after a freezing-and-thawing cycle, as discussed by Powers [*13*]. Either weight loss due to sloughing or reduction in resonant frequency may occur without accompanying length change.

Kesler and Higuchi [*36*] have reported on the relation between compressive strength and sonic properties without, however, consideration of how either of them may indicate freezing-and-thawing damage.

The measurement of the velocity of propagation of a sonic pulse through concrete has been used to explore deterioration of concrete, especially in field structures. This subject is explored at more length in another paper in this publication.

EFFECT OF DEICING CHEMICALS

The use of chemicals, mainly calcium or sodium chloride, to clear ice from highways in the winter has increased tremendously in recent years. In many cases this use has led to serious damage to concrete pavements and other structures. The damage usually takes the form of more or less extensive scaling or spalling which, in addition to being unsightly and in itself destructive of the integrity of the concrete, makes it more vulnerable to other deleterious factors. Water can more easily enter, and the chance of further freezing-and-thawing damage is increased. Roberts and Vivian [*37*] have reported on the widening of expansion cracks in mortar by salt solutions.

The phenomenon of deicer damage has been studied by a number of investigators but is still not well understood. Verbeck and Klieger [*38*] found that, of a number of agents which they investigated, all those that were effective as deicers also produced scaling. Also they observed that relatively low concentrations of deicer produced more scaling than did higher concentrations or absence of deicer. These facts led them to conclude that, although chemicals are involved in deicing, the mechanisms by which deicing chemicals cause damage is physical. Lawrence and Vivian, however, report a severe reaction causing disruption when strong calcium chloride solution comes in contact with mortar [*39*]. The disruption is stated to be similar to that caused by sulfate attack and is attributed to the formation of chloroaluminate.

Several investigators have emphasized that the production of dense, high-quality concrete, with adequate entrained air and with adequate curing and a period of drying before the first application of deicing agents, are essential in preventing damage [*40–42*].

Various surface coatings or treatments have been tried as protectors of concrete from the effects of deicing chemicals, and conflicting results have been reported. Hussell [*43*] found that silicones did not increase the resistance of nonair-entrained concrete to an acceptable level, while Klieger and Perenchio [*44*] found indications that silicones might be detrimental. In studies by Grieb and Appleton [*45*] linseed oil coatings were found to be of some benefit in protecting the surface of nonair-entrained concrete but did not increase the resistance of air-entrained concrete to the same degree. Grieb, Werner, and Woolf [*46*] found that: (1) all the air-entraining agents they used were effective to different degrees in delaying the start of scaling, (2) scaling was less pronounced when specimens were cast on a sand base rather than on a watertight metal base, (3) scaling was less when low-alkali cement was used, (4) concrete stored in room air at 50 per cent relative humidity had better resistance to scaling than moist cured concrete, (5) curing materials applied shortly after the concrete was cast were of some benefit in delaying the development of scaling, (6) vacuum treatment of the concrete surface increased resistance to scaling, (7) protective surface treatments applied 14 to 28 days after casting delayed but did not prevent the development of scaling, and (8) silicone and latex emulsion admixtures were effective in preventing scaling but had some more or less detrimental side effects. They showed some evidence that fly ash as a partial replacement for cement in certain amounts might be beneficial, with low carbon fly ash being superior to fly ash with a high carbon content. As further preventive measures they list elimination of waterproof coatings or sheetings on the subgrade, reduction of the water-cement ratio, permitting the concrete to dry before it is first frozen and, for concrete placed shortly before freezing is anticipated, minimizing wet curing.

Care in the selection of deicing chemicals needs to be exercised. Deicers containing ammonium sulfate or ammonium nitrate should never be used [*47*]. These chemicals react with lime in the cement to produce soluble calcium salts and cause rapid deterioration of concrete.

OTHER WEATHERING PROCESSES

Other weathering processes which can cause deterioration of concrete include heating and cooling and wetting and drying. Both of these processes produce volume changes, and if the volume changes are excessive or if large differences between the thermal or moisture dependent properties of different constituents of the concrete exist, damage to the concrete may result. Pearson [*48*] has reported on a case where deterioration was due to aggregate with an abnormally low coefficient of thermal expansion which caused large strains in the concrete when temperatures dropped suddenly. No cases of abnormally high coefficients of expansion have been reported. Most work in this field has been related to the effect of aggregates with different coefficients of thermal expansion on freezing-and-thawing resistance [*49,50*].

The effect of wetting and drying on concrete has been studied more than heating and cooling. Mitchell [*51*] found that the thermal coefficient of neat cement depended on the degree of saturation and that the coefficient of thermal expansion of concrete can be determined from the weighted averages of the coefficients of thermal expansion of the various constituents if the correct moisture effect can be determined. Snowdon and Edwards [*52*] found that concretes made with aggregates which had high drying shrinkage expanded progressively with cycles of wetting and drying.

In the same work that included the study of the effect of wetting and drying cycles, Snowdon and Edwards found that failure on exposure to the weather tended to increase when highly shrinkable rock was used as coarse aggregate, although a firm relationship between weathering and rock shrinkage was not established. For one limited group of tests, damage by weathering tended to decrease with increasing fineness of the fine aggregate. The greatest damage occurred with a combination of high-shrinking fine aggregate and nonshrinking coarse aggregate.

CONCLUSION

A discussion necessarily as brief as this one on the subject of Resistance to Weathering should not close without mentioning some other investigations and publications which are useful sources of information in this important field. The excellent report of ACI Committee 201 on Durability of Concrete in Service [*53*] is quite comprehensive and covers a number of aspects of durability not dealt with in this discussion since they are the subjects of other papers in this publication. Highway Research Board Committee MC-B1 has published a comprehensive annotated bibliography on the subject of Durability of Concrete: Physical Aspects, which covers the literature on the subject through 1956 and which is now being updated to include material through 1964 [*54,55*].

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Hardened Concrete

RESISTANCE TO CHEMICAL ATTACK

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Fortunately most concrete in service is not subjected to chemical attack. Concrete is generally less able to resist successfully corrosion of this kind than disintegration by other forces. By chemical attack is meant leaching and acid or sulfate attack.

Although concrete is often disfigured by deposits of efflorescence [*1*][2] as lime water leach reaches outside surfaces, very little good-quality concrete has actually been destroyed or made unserviceable by leaching of lime from internal or external surfaces. In addition to good workmanship, probably the best initial correctives are either an ample cement content with a low-water content and water-cement ratio or a cementitious mixture of portland cement and a good, active pozzolanic material to minimize permeability.

Acid attack is commonly encountered in sewers above the flow line, on floors of food processing plants, and elsewhere. In the case of sewers, attack on concrete is by sulfuric acid; little can be done about it directly but much can be done to prevent it by proper sewer design. The lactic and acetic acid attacks are milder and can be minimized by good workmanship, good concrete, and a good pozzolanic material added to the cement to make the lime less subject to attack by acids.

Sulfate attack can completely disintegrate average concrete made with ordinary cement in a very few years. Many field structures have become unserviceable from disintegration by sulfate salts, usually of sodium, magnesium, or possibly calcium, which exist naturally at varying intensity in many soils, shales, and ground waters. This chemical attack can be controlled by using at least six sacks of Type V sulfate-resistant cement per cubic yard of concrete at the lowest practicable water-cement ratio.

With this summary of the general problem of chemical attack and the means for producing concrete to resist it, this report will proceed with a more detailed discussion of the significant aspects of how these chemical actions damage concrete, what can be done to combat them, and in some cases what tests can be used to measure the degree of attack or the success of corrective measures.

Leaching

Hydrated lime is one of the compounds formed when cement and water combine in concrete. This lime is readily dissolved by water, particularly if the water is lime-free and contains dissolved carbon dioxide. Snow water in mountain streams and reservoirs is often particularly aggressive because it is unusually cold (calcium hydroxide is more soluble in cold than in warm water), pure, and consequently lime-hungry, and, like most surface water, contains carbon dioxide.

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

This produces a mild carbonic acid solution that has a higher capacity for dissolving lime than does pure water without it [*1,11,14,16,17,18,19,22*].

As a result, inside surfaces of concrete conduits, flumes, and canal linings develop a sandy appearance from having the cement matrix leached and weakened by contact with these lime-hungry waters. Scandinavians report [*4,31*] serious attack and decay of this kind in dams and conduits for relatively lime-free water; however, the later report [*4*] indicates that its severity was due to the poor quality of concrete at these locations.

FIG. 1—Lime-hungry or slightly acid water has leached surface mortar and exposed small aggregate on surfaces of this siphon inlet.

Attack by snow water has been noted in power flumes and lined canals carrying water from the California Sierras (see Fig. 1). An outstanding California example is the 15-mile Tiger Creek flume of the Pacific Gas and Electric Co. A few years after construction in 1930, the surface was found to be considerably roughened, and it was feared further deterioration would seriously reduce its capacity. After a trial length that had been coated with well-cured, troweled gunite using ordinary portland cement showed that it too was being attacked, the entire flume interior was painted with a coal-tar-base paint which had performed best in tests of many paints and coatings previously tried in the flume. Even with this treatment, repainting was required after eight years' service.

Objectionable results of leaching are not confined to surfaces in contact with pure mountain water. Exposed surfaces of tunnel linings, retaining walls, abutments, and other structures, where ground water has access to the opposite side, are often disfigured by lime deposits. These are formed by water that has come through the concrete, either along cracks or joints or through porous areas, taking lime into solution and becoming saturated with it. At the surface the solution absorbs carbon dioxide which reacts with the calcium hydroxide and causes precipitation of a white deposit of calcium carbonate. Although unsightly, this kind of leaching rarely has detracted significantly from the basic serviceability of such structures made of good concrete.

Investigators have attempted to measure the influence of cement composition and the use of pozzolanic material on the degree of leaching which may occur. An unpublished report by Stenzel for the Metropolitan Water District of Southern California in 1936 found for typical Types I, II, and V cements that "the leaching effect is similar in nature and magnitude for all the cements studied. The effect [for a portland-pozzolan cement] is, however, definitely less than for the portland-cement group, even when the reduced actual cement content (80 per cent) is taken into consideration." In 120 days of leach tests, more than 20 per cent less solids were dissolved from the mortar made with the 80-20 portland-pozzolan cement. Stenzel further concluded: "Since the laboratory experiments indicate that the actual quantity of cement dissolved is of a low order of magnitude, it may be said that failures from this source are not likely to occur before those due to weathering and other causes." He added: "As the lime is exhausted from the rich surface layer of the concrete, it is to be expected that the leaching process will greatly subside" and be further inhibited by slower diffusion.

From these observations and other studies of the problem, it would seem appropriate to recommend the following ways to minimize leaching:

1. Use of:
   (*a*) aluminous cements,
   (*b*) portland - blast furnace slag cements,
   (*c*) portland-pozzolan cement with 20 to 30 per cent of a good pozzolanic material that is strongly active in combining with lime to form insoluble lime silicates, or
   (*d*) "low-lime" portland cement with less tricalcium silicate than dicalcium silicate.
2. Use of sufficient cement and a low water-cement ratio to insure good dense concrete together with air entrainment, to minimize permeability and capillarity, inhibit diffusion, and retard dissolving of cement increasingly after the surface has been leached, particularly in water channels.
3. Design and use of contraction and construction joints that are watertight and frequent enough to prevent intermediate cracking.
4. Proper control of concrete mixes, placing procedures, and curing so that hardened concrete free from permeable imperfections is insured.
5. When practical, provision for drainage facilities necessary to prevent water from standing behind structure walls.
6. Consideration of a provision for a protective coating of durable and effective surface sealing material, if such can be found and reliably applied. (See Portland Cement Association Bulletin "Effect of Various Substances on Concrete and Protective Treatments, Where Required.")

ACID ATTACK IN SEWERS

Problems of acid attack above water levels in sewers are usually assumed to be the result of sulfuric acid formed from hydrogen sulfide gas, generated by bacteria from sulfur compounds in the sewage, which rises and combines with oxygen and with moisture condensed on upper surfaces of the sewer conduit [7]. This is not a direct attack of acid in the sewage. Records indicate that the usual run of sewage and waste water averages very close to neutral, fluctuating only slightly above and below a pH of 7.0. Predominance of certain industrial wastes in certain locations may change this significantly and present a special problem.

For most sewers, however, since the flow itself is not aggressive, problems of acid corrosion in the crown portions are best solved by design or operating modifications that result in the sewers running full or with ventilation at higher velocities. Lower temperatures also tend to reduce the production of acid-forming hydrogen sulfide gas [7]. When sewage is stagnant or moves slowly, bacterial creation of the sulfide is often too rapid to be oxidized by air dissolved in the sewage.

FIG. 2—Disintegration of concrete in the crown of a sewer is caused by sulfuric acid formed as hydrogen sulfide gas rising from the sewage and combining with oxygen and moisture on the concrete.

Unfortunately many existing systems are not amenable to such changes, and serious problems of acid corrosion exist in their crown portions (see Fig. 2). The fact should be faced that, in such aggressively corrosive situations, portland-cement concrete is not the proper material to use since none of the various compositions of portland cement are resistant to acid corrosion. Other materials or protective coverings or coatings should be employed.

It is reported [*27,28*] that calcareous aggregate tends to neutralize acids in sewage and so reduces the rate of deterioration of concrete even though it will still be attacked. Hence, use of limestone or dolomite aggregate may be a desirable measure where concrete must be used in sewerage for reasons of design or otherwise.

F. L. Smith and J. E. Backstrom, in an unpublished report for the U. S. Bureau of Reclamation in 1952, found that in 5 per cent solutions of sulfuric acid with a pH of 0.2 both plain and air-entrained concrete lost strength rapidly, and lost weight, from progressive surface disintegration, amounting to approximately 50 per cent in 12 weeks' immersion. Actually, 5 per cent is much stronger than the acid solutions attacking sewers, but it is considered that this concentration provides a reliably indicative accelerated test of relative resistance to acid attack. The tests were made on 3 by 6-in. cylinders of concrete having a water-cement ratio of 0.52, $\frac{3}{4}$-in. max aggregate, and a slump of about 2.7 in.

TABLE 1—COMPARISON OF UNTREATED CONCRETE AND OCRAT-CONCRETE.

	Untreated		Ocrated	
Removed In Abrasion Test, $\text{in.}^3/\text{in.}^2$; Aged 6 Weeks.....	0.061		0.040	
	Tension Specimen	Compression Specimen	Tension Specimen	Compression Specimen
Portland cement and Rhein sand 0 to $\frac{1}{8}$-in.; mix 1:3.5 by weight, plastic; water-cement ratio = 0.68; cured 3 days, then air dried at 6 weeks, psi................	711	2930	1408	6045
Portland - blast furnace slag cement, 30 per cent max slag; water-cement ratio = 0.36; mix 1:5 by weight, earth moist; strength at 6 weeks, psi.......................	384	1252	754	3001
Dycherhoff white cement; mix 1:3 by weight, same materials:				
Strength at 6 weeks, psi..................	569	3300	1166	7368
Strength at 8 weeks, psi..................	626	3357	1180	8335
Strength after 6 weeks immersion in acids and sulfate solutions after ocrating at 28 days, psi:				
Sodium sulfate solution:				
Portland cement......................	1152	4025	1735	6429
Portland - blast furnace slag............	1195	4395	1579	5206
10 per cent acetic acid solution:				
Portland cement......................	441	1920	1565	5362
Portland - blast furnace slag cement.....	484	1721	1422	4893
Dycherhoff white cement...............	455	2091	1380	4992
Lactic acid:				
Portland cement......................	427	1465	1408	6472
Portland - blast furnace slag cement.....	612	2176	1408	4964
Dycherhoff white cement...............	583	2247	1394	4592
5 per cent hydrochloric acid:				
Portland - blast furnace slag cement.....	destroyed after 5 hr		1707	7069

Investigators are continuing the search for materials or treatments that will make concrete a suitably resistant material to chemical attack. ASTM Test for Chemical Resistance of Hydraulic-Cement Mortars (C 267) describes a method of test which is essentially a description of methods used by most investigators. Results of these many research tests have identified materials and mixes that have not been resistant to various forms of chemical attack in service. Such methods are considered to be significant because they have pointed the way to other materials and practices which are giving far better service.

Many investigators have used immersed-storage and flow-over-the-surface tests with specimens of various materials, mixes, sizes, and shapes being exposed to various acid solutions for intervals between which surfaces were brushed to remove loosened or softened material [*8,13*]. Such tests, when suitably representative of the nature of attack, gave a significant indication of the degree of corrosion and the degree of resistance that may be expected. It is best that results be judged and compared from depth of scaling rather than from loss in weight, since loss varies with volume of the test specimens, whereas depth does not.

A German investigator has reported initial encouraging results with a process called "Ocrat-Concrete" [*21*]. The treatment[3] consists of subjecting the concrete in a vacuum to the action of silicon tetrafluoride gas (SiF_4): $2Ca(OH)_2 + SiF_4 = 2CaF_2 + Si(OH)_4$. When given proper time in the treatment, the fluoride gas penetrates to some depth and provides more than a thin shell of resistance to acid attack.

So far this treatment is applicable only to precast units, and it has the drawback of being so toxic that exceptional care would have to be exercised to protect workmen. However, the results of tests are encouraging (Table 1).

OTHER ACID ATTACK

Common among other acid attacks are lactic and acetic acids in dairy and fruit products spilled on concrete floors of food processing plants. Although attack from these products is comparatively mild, it is persistent and can result in softening a working floor so that it wears rapidly and becomes uneven and unsatisfactory for smooth operation of wheeled trucks, dollies, and carts. Another common area of acetic acid attack (pH 3.4 to 3.9) is due to silage in concrete silos [*8,13*].

Swedish moor water with pH values as low as 4.5 has been destructive to somewhat porous concrete pipe. The water contained considerable aggressive carbonic acid [*4*]. One English moor water was made weakly sulfuric acidic by contamination from a polluted industrial atmosphere and from pyrites in the soil [*9*]. Another soft English moor water was quite acidic, averaging a pH of 4.4, probably due mainly to organic acids washed from peat, sometimes collectively described as humic acid. Exposed to a flow of this water for 4 years, 4-in. concrete cubes lost considerably more strength than would be indicated by their 4 per cent loss in weight [*5*]. These observations suggest that need for precautions against acids may exist and must be recognized. Not all aggressive surface waters are sulfates, although they are the most common.

The weakening effect of these acids on portland-cement mortar is noticeable in the strengths reported in the German data shown in Table 1 [*21*]. Among these same tests it may be noted that the portland - blast furnace slag cement mortar was improved in strength during 6 weeks of storage in these acid solutions. This cement contained not over 30 per cent slag. In 1927 Schlyter [*24*], recommended slag or aluminous cement for better acid resistance.

The American Concrete Pipe Assn. [*3*] reports results from tests in which pipe concrete with Type I cement was immersed for 9 months in acetic acid (pH = 2.5) after 6 months of water curing. Clearly superior resistance was found in specimens in which 25 per cent of the cement had been replaced with a calcined opaline shale pozzolan. This report suggests, however, that reliance should not be placed on serviceability of such portland-pozzolan cement concrete if pH values of attacking acids are less than 5.0.

Super-sulfated cement is a mixture of granulated blast furnace slag with some anhydrite (calcium sulfate) and a small addition of lime or portland cement. This cement is being investigated as a means

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[3] German Patent No. 849,225, Dec. 5, 1951.

FIG. 3—This World War II air base barrack floor in a desert location showed distress in a few years from sulfate attack.

FIG. 4—Corrosion by sulfates has completely disintegrated lower parts of this drop structure in a western canal.

for creating a concrete resistant to acids, especially in sewers, but tests by contemporary investigators give no indication that super-sulfated cement is resistant to acid attack [*5*]. Most investigators report best results with it in resistance to sulfates and sea water [*2,7*] and imply, by omission of the subject, that it contributes nothing to acid resistance.

Basically, no portland-cement concrete is acid-resistant. However, there is great difference in the degree of resistance obtained in the wide range of cementing materials and workmanship used. When the most resistant cements are used in good, well-placed, impermeable concrete in well-designed and well-drained structures, much better serviceability may be expected. In detail, the same recommendations made for resistance to leaching are suggested for best resistance to mild acids. However, where strong acid attack is expected, other materials should be used, or at least a protective surface treatment of proved effectiveness should be provided.

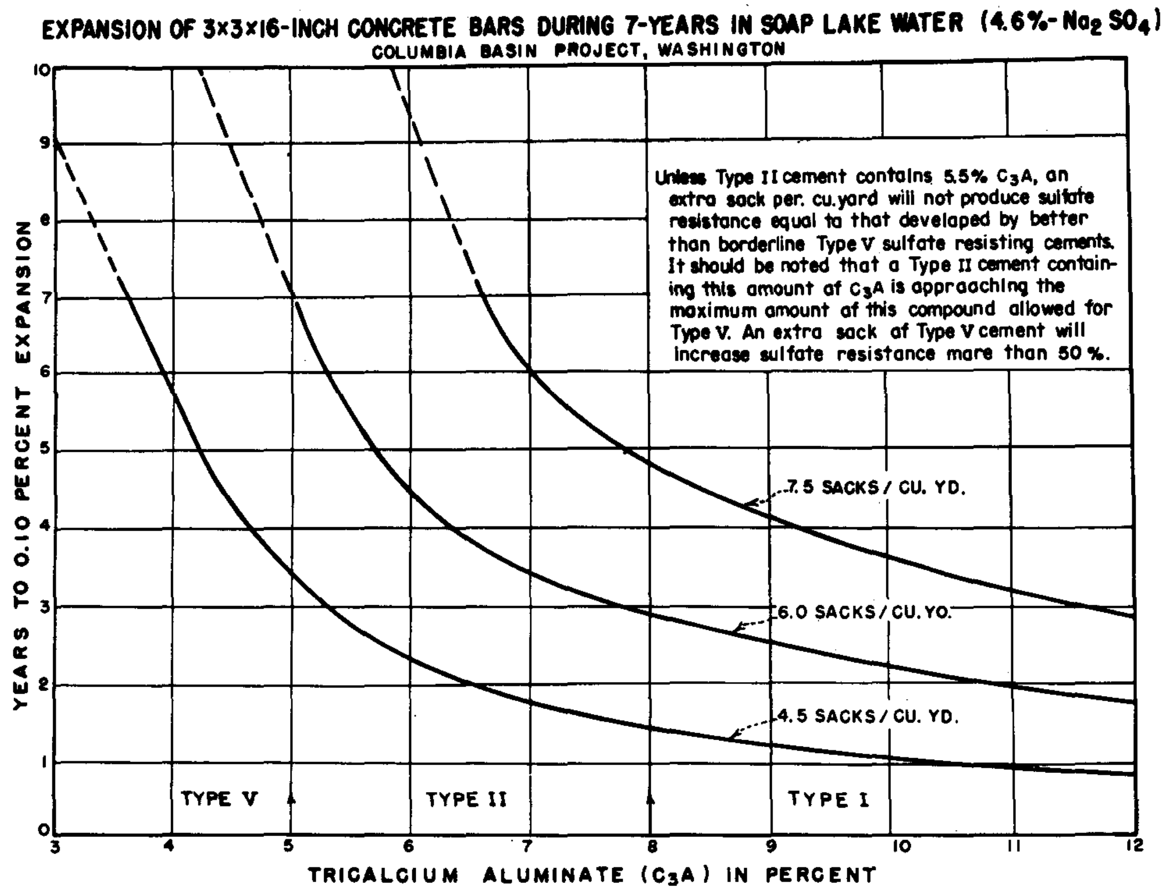
FIG. 5—Concrete under ground was intact, but rising sulfate solutions caused exposed surfaces to spall away as salt crystals developed in concrete pores as moisture evaporated.

SULFATE CORROSION

Some of the most spectacular chemical attacks encountered in concrete have been a result of sulfates in adjacent soil and ground water (see Figs. 3–5). The degree and rate of this attack vary. These attacks increase as the concentration of sulfates in surrounding water increases and decrease as the amount of dry exposure increases. Dry concrete in dry sulfate-bearing soils will not be attacked. Where saturation is continuous in strongly sulfate-bearing ground water, the attack will be rapid and severe. It is even more severe where saturation and drying are frequently alternated. Average concrete with Type I cement has been completely disintegrated in only a very few years when severely exposed in drainage pipe, floors, and lower parts of canal structures.

The most common and aggressive sulfates are those of sodium and magnesium, but some structure and canal locations are in soil containing appreciable amounts of calcium sulfate (gypsum). Because gypsum is relatively insoluble, sulfate attack on concrete may be negligible. However, if ground water movements are sufficiently sluggish, it is possible for it to become a saturated solution of gypsum which contains 1100 ppm SO_4. This is a concentration that is capable of a considerable degree of sulfate attack, although it is likely to be at a slower rate than with sodium sulfate because it has been determined that calcium sulfate reacts with hydrated tricalcium aluminate but not with hydrated lime or the hydrated silicates.

Both the manner of sulfate corrosion and one means of retarding it are mentioned in the following statement of Bogue [*2*]: "Since calcium sulfoaluminate forms most readily in concentrated solutions of $Ca(OH)_2$, and since the disintegration of cements in sulfate solutions is due chiefly to the formation of that salt, the presence of active silica will retard the formation of the sulfoaluminate and thus delay the disintegration of the structure." Presumably this implies that active silica or pozzolan is helpful in reducing the severity of sulfate attack by combining with $Ca(OH)_2$ and making much of it insoluble.



Unless Type II cement contains 5.5% C_3A, an extra sack per. cu.yard will not produce sulfate resistance equal to that developed by better than borderline Type V sulfate resisting cements. It should be noted that a Type II cement containing this amount of C_3A is approaching the maximum amount of this compound allowed for Type V. An extra sack of Type V cement will increase sulfate resistance more than 50 %.

FIG. 6—The lower the percentage of tricalcium aluminate in the cement, and the richer the mix the better will be the resistance of concrete to sulfate attack.

A less severe effect has been noted in some exposures where underground concrete was unaffected, but the surface of the concrete immediately above the ground was pitted and scaled away (see Fig. 5). By a wicking action, sulfate solution rises in the concrete. In arid climates, as it evaporates from concrete in surfaces just above the ground, crystals of sulfoaluminate and sulfate develop in pores under the surface with sufficient force to flake off the concrete [*15*].

The widespread nature of the problem and the vigor of sulfate attack stimulated a search for means to combat it, and as early as 1920 D. G. Miller commenced his lifelong study of this problem with P. W. Manson. This is summarized in their University of Minnesota Technical Bulletin No. 194, "Long-Time Tests of Concretes and Mortars Exposed to Sulfate Waters." Other investigators, including the Portland Cement Assn., the Bureau of Reclamation, and the Metropolitan Water District of Southern California [*20*], similarly arrived at the same basic conclusion: that the lower the percentage of tricalcium aluminate in the cement, the better would be the resistance of concrete to sulfate attack. Typical results of tests by the Bureau of Reclamation are plotted in Fig. 6.

Essentially, these tests involved exposure of concrete specimens immersed in sulfate waters. Specimens ranged from half briquets of standard 20-30 Ottawa sand mortar to 6 by 12-in. concrete cylinders and lengths of commercial precast concrete pipe. These test specimens contained a wide variety of cements, pozzolanic materials, and admixtures in mortars and concretes of varying richness and curing. Effects of curing, temperature, and exposure to drying were determined. Concentrations of sulfate were varied, and effects of both sodium and magnesium sulfates were noted. Lack of resistance was evaluated: from expansion, 0.5 per cent being considered as complete failure; from loss of strength compared to that of specimens stored in plain water; from loss in dynamic modulus, complete failure being a loss of 40 per cent; from significant loss of weight; and from obvious visual failure.

In these tests, made under normal exposure conditions, it did not take long to identify unresisting concretes, but it took many years to prove the relative merit of comparatively resistant materials and treatments. Consequently, the Bureau of Reclamation laboratories in Denver, Colo., are using currently an accelerated sulfate test for evaluating sulfate resistance of concretes. This test produces results comparable to those of the 70 F continuous soaking test in approximately one-sixth the time. In this test 3 by 6-in. specimens are alternately soaked in a 2.1 per cent solution of sodium sulfate at about 73 F for 16 hr and then dried in air at 130 F for 8 hr. The criterion of failure in this test is established as being a loss of 15 per cent in dynamic modulus or an expansion of 0.2 per cent. At this point, internal disruption has definitely started, and complete failure of the concrete, indicated by a 40 per cent reduction in dynamic modulus or an expansion of 0.5 per cent, is imminent. Indications from this accelerated test have been consistent with long-time results well established during the past 25 years.

The Subcommittee on Sulfate Resistance of ASTM Committee C-1 Cement, has developed an expansion test which in 14 days reflects with fair reliability the resistance to sulfate attack which may be expected from the cement used to make up the test bars. The method is described in ASTM Test for Potential Expansion of Portland Cement Mortars Exposed to Sulfate (C 452). Prompt sulfate action is generated by mixing sufficient powdered calcium sulfate with the cement for it to contain 7.0 per cent SO_3. Some feel that the results of this test can be used best only as a supplemental indication to compound composition requirements mentioned below in item 3, since on occasion cements having 6 to 9 per cent tricalcium aluminate have been indicated by this test to be comparatively sulfate resisting.

These high values of tricalcium aluminate raise serious doubts for sulfate resistance. Whenever sulfate resistance of a comprehensive range of cement compositions has been obtained and tabulated against the per cent of tricalcium aluminate in each composition, such as in William Lerch's Table 1, "Summary of Sulfate Resisting Performance of Cements in the Long Time Study of the Portland Cement Assn.," reported in Ref. *26*, and in Miller's Table 6, "Summary of 122 Commercial Cements," p. 33 of Ref. *25*, and in many other such summaries, the conclusion is inescapable that low tricalcium aluminate content correlates positively and most reliably with greatest sulfate resistance.

Actually, in the long-time tests, except for differences in the effects of pozzolanic materials and admixtures, it has been found that the time required to develop an expansion of 0.10 per cent correlates quite accurately with ultimate resistance performance. Information in Fig. 6 is presented on this basis. Results of the long-time exposure tests have proved quite significant in that concrete in service, made with the cements, mixes, and curing found most resistant in these tests, has resisted sulfate attack far better than concrete made with most cements.

Although the low tricalcium aluminate in Type V cement is without doubt the most important factor in improving resistance of concrete to sulfates (Fig. 6), there are several other provisions and precautions that will contribute materially to sulfate resistance:

1. The concrete should have an ample cement content. A water-cement ratio of not more than 0.50 should be used. For severe exposure an additional sack of Type V sulfate-resistant cement will increase resistance at least 50 per cent as shown in Fig. 6. It is also evident that sulfate resistance, equal to that provided by Type V cement with 4.5 per cent tricalcium aluminate, is not obtained by using an additional sack of Type II cement per cubic yard unless the Type II cement contains less than 5.5 per cent of tricalcium aluminate.

2. An active pozzolanic material may be substituted for 15 to 30 per cent by weight of cements that are relatively high in tricalcium silicate [*6*]. The lesser percentages of pozzolanic material should be used if the pozzolan has a high-water requirement. Pozzolan effectiveness is substantially greater if several months of curing can be provided prior to sulfate exposure, since its combination with the lime is slow and will stop without moisture. The sulfate resistance of low tricalcium aluminate Type V cement is not benefitted and is sometimes decreased [*29*] by use of pozzolan. Resistance of concrete with Type V cement is increased most effectively and noticeably by an increase in cement content of 10 to 20 per cent and a corresponding reduction in water-cement ratio.

3. A low-lime and low-aluminate cement, having less than 50 per cent tricalcium silicate and less than 12 per cent tricalcium aluminate plus tetracalcium aluminoferrite, in which less than 4 per cent is tricalcium aluminate, makes the most sulfate-resistant portland cement. Specifications for sulfate-resistant cement having these compound limits were written in 1934 for work in southern California [*20*].

It was felt that when long-range durability of concrete is at stake in an aggressive environment it is not as important to require composition limits that most manufacturers can meet, as it is to specify limits that will provide all the sulfate resistance that can be put into the cement. This is advisable even at a somewhat higher premium, perhaps to only one mill, if there is only one mill which can make the cement to those safer limits.

4. Precast units such as concrete pipe and block can be made appreciably more resistant by a period of several weeks of drying following good curing, as indicated by the data in Fig. 7. Presumably carbonation occurring during this period contributes to improved sulfate resistance, as it also improved resistance of leaching, by reducing permeability.

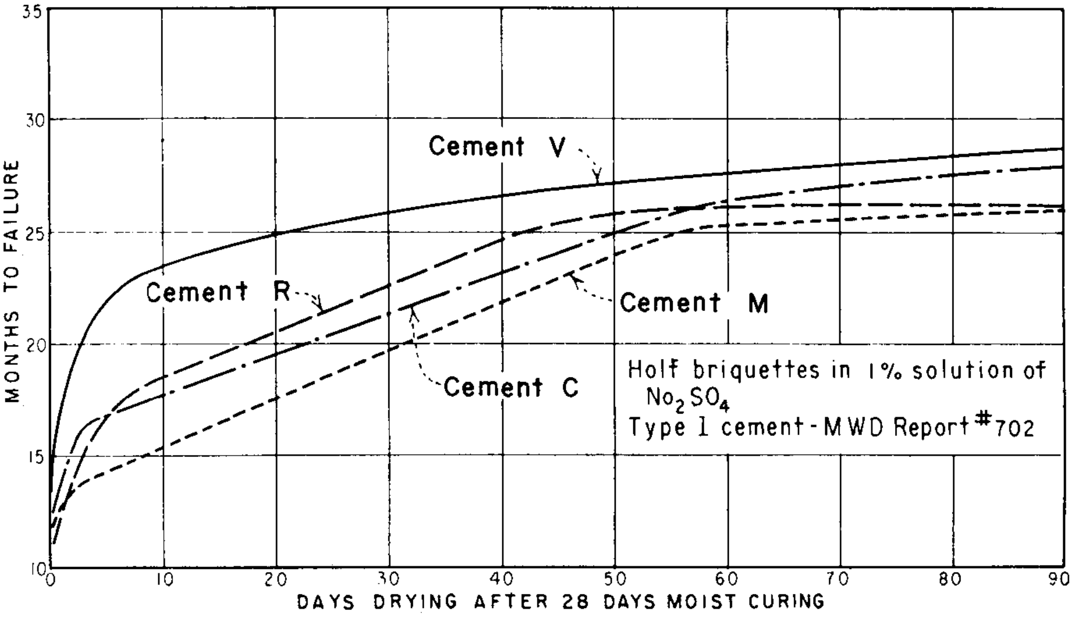


FIG. 7—Resistance to sulfate corrosion is materially increased by drying concrete several weeks after good curing.

5. As indicated by Miller [*25*], steam curing at 350 F or above greatly improves the resistance of specimens, including those made with ordinary portland cements.

6. Calcium chloride reduces sulfate resistance regardless of the type of cement used.

ATTACK BY OTHER CHEMICALS

Most widespread among attacks by other chemicals is the scaling of concrete pavements and sidewalks caused by the use of sodium or calcium chloride to clear ice and snow in freezing weather. Actually this scaling is not primarily the result of chemical attack as evidenced by the considerable freedom from scaling obtained when concrete is amply air entrained. In such a rapid physical attack, these salts have the effect of increasing the severity of freezing-and-thawing cycles and, unless concrete is unusually resistant to such weathering, surface disintegration results. The physical actions involved in such weathering may be supplemented by leaching because of the greater solubility of calcium hydroxide in calcium or sodium chloride solutions, or by the formation of chloroaluminates under wetting and drying conditions. Presumably by such chemical actions calcium chloride has caused disintegration of concrete where brine has dripped from refrigerating equipment and where no freezing was involved.

Since a pavement is disintegrated mainly by cycles of freezing and thawing, experience has shown that this result is largely prevented by using 5 to 7 per cent of entrained air in the concrete, by keeping slumps low, and by avoiding overmanipulation in finishing which brings water to the surface and works entrained air out of the concrete.

Sea water is mentioned mainly to point out that most concrete failures in this connection have not been due to chemical attack of the sea water so much as to porosity which enhanced leaching and expansive corrosion of reinforcing bars, and to freezing and thawing at tide and wave levels [*7,11,16,17,18,19,30*]. In one notable example in southern California, alkali-aggregate reactivity contributed to failure. However, sulfate constitutes a little over 0.25 per cent of sea water, a concentration that is regarded as sufficiently severe when found in ground water to warrant use of Type V sulfate-resistant cement and other precautions previously mentioned to secure sulfate resistance. The additional precautions of good workmanship, air entrainment, low water-cement ratios, and ample cover over reinforcing steel should largely offset the other recognized causes of failure of concrete in sea water. European literature speaks favorably of the performance of aluminous and slag cement and of portland-pozzolan combinations in sea water exposure [*9,10,12*].

With increasing attention to conversion of sea water, plants of larger capacity may utilize as much concrete as possible structurally. This possibility has raised the question of the effect of hot sea water and of hot distilled water on concrete durability. The California Department of Water Resources has prepared a series of test specimens for exposure under identical conditions in a sea-water conversion plant and the Bureau of Reclamation is conducting pertinent tests for the Office of Saline Water of the U. S. Department of the Interior.

Sulfur water is an occasional source of concrete deterioration according to reported tests [*14*]. Evidently, aggressive sulfate salts exist in this water and are responsible for the attack. Arrangements were made to submerge 6 by 12-in. cylinders of various cements and mixes in continuously flowing water at the job site for comparison with similar cylinders stored in fresh water. Results indicated that "the special cements—Lumnite, Trass, and Super," or mixes with fly ash or pumicite substituted for part of the cement were even better than rich mixes and low water-cement ratios. Appropriate warning is given that heat generated by Lumnite cement in confined massive placements may be formidable and objectionable unless precautions are taken to care for it.

RELATION OF AGGREGATES TO RESISTANCE TO CHEMICAL ATTACK

Except in its relation to resistance to chemical attack, it is not the purpose of this paper to discuss the influence of aggregate on concrete durability. This is covered in greater detail in other papers in this symposium. Indications from available records of experience are that aggregate is secondary in responsibility for lack of resistance to chemical attack. Although some aggregates may be somewhat vulnerable, as limestone, for example, is vulnerable to acid, failure of concrete to resist chemical attack is primarily a failure of the cement paste. If the paste can be made resistant, the concrete will be resistant and serviceable, if the aggregate otherwise makes serviceable concrete.

CONCLUSIONS

This brief survey indicates that there are many resources in materials and benefits from careful workmanship which can be marshalled quite effectively against many kinds of chemical attack on concrete in service. There are, however, certain exposures to which concrete is inherently unresistant, and no means are now known by which it can be made satisfactorily resistant. These cases should be recognized in advance, and other materials of proved resistance should be used.

Acknowledgment:

The author is indebted to A. B. Crosby of the staff of the U. S. Bureau of Reclamation Concrete Laboratory for his considerable assistance in reviewing pertinent technical literature. Recognition is made also of valuable work reported by contemporary investigators mentioned in the text and in the references.

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STP169A-EB/Apr. 1966

Hardened Concrete

RESISTANCE TO HIGH TEMPERATURE

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In evaluating resistance to high temperature, essentially every attribute and property of concrete merits consideration other than that of the concrete's resistance to freezing and thawing. These pertinent factors include items such as the quality of the ingredients, the design of the concrete mix, extent of curing and drying, volume change, thermal insulation, compatibility of aggregates and cement, carbonation, porosity, wetting and drying, and not least of all, strength. This complexity is real in spite of the fact that concrete is unquestionably accepted as the one structural material which has marked resistance to fire; consequently, any type or class of concrete is acknowledged by most laymen to be fire resistant. The specialist however, breaks the general category of concrete down into heavyweight, normal weight, lightweight, and ultra lightweight concretes, and further yet, into those made with natural or manufactured aggregates and with various kinds of hydraulic cement; he also gives due consideration to degree of exposure as well as ultimate use and purpose of the structure involved. A one-shot exposure to heat or fire is contrasted with repeated or continued exposure. Only a brief review is possible of the phenomena and facets whereby concretes attain these attributes. Further information may be obtained from the references.

EFFECT OF CEMENTS

ASTM specifications cover a range of various portland cements, as well as other hydraulic cements such as slag, natural, and masonry. In addition to these, a high-alumina cement is available on the market; amongst its several uses, high-alumina cement finds specific application in the field of refractories. The characteristics and performance of concrete employing high-alumina cement [*1,2*][2] are considered to be outside the scope of this paper inasmuch as these concretes differ considerably from those made with portland-type cements.

The portland cement matrix in hardened concrete begins to dehydrate below 500 F; given enough time it will dehydrate completely at about 1000 F, and at 2000 F it will dehydrate in a very short time. Tests reported by Philleo [*3*] have shown that hardened cement paste ***upon initial heating*** exhibits normal expansion with a rise in temperature up to about 300 F; above this, it shrinks or contracts appreciably with its first rise in temperature up to 1000 F. This linear contraction or shrinkage due to initial dehydration, particularly in the range 500 to 750 F, has been shown to progress at the rate of about 40×10^{-6} per deg F rise in temperature; this would indicate that cement paste subjected to high temperatures must undergo considerable cracking when held in restraint. In the

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

lower and normal range of temperature (below 212 F) hardened cement paste evidences a coefficient of thermal expansion in the range 6 to 12 $\times 10^{-6}$ per deg F [*4*,*5*].

In addition to the thermal and shrinkage changes which occur with increase in temperature, consideration must also be given to the fact that the compounds in hydrated cement are susceptible to change. Hydrated cement paste could conceivably be heated high enough to approach in composition the cement clinker from which it was first ground. Changes in chemical composition occur such as those at about 1000 F where calcium hydroxide is decomposed and at 1500 F where calcium carbonate is transformed. These are simple changes which occur in cement paste, but, even here, one may note that calcium oxide will be one of the compounds resulting from high enough heat; volume change related to this compound alone during calcination or subsequent rehydration and carbonation or both is enough to cause distress and loss of strength. Concrete subjected to a sustained temperature of 1200 to 1500 F can usually be taken apart with one's fingers. Thus it is that some authorities lean toward the use of portland cements which upon hydration release least amounts of calcium hydroxide; for this reason, preference is sometimes given to the use of slag cement or of portland blast-furnace slag cement [*6*]. However, sufficient data are not available to support such distinctions among portland cements or between portland cement and the slag cements.

EFFECT OF AGGREGATES

As to aggregates, there is no standard specification setting forth various qualities which this ingredient should possess and those which are necessary or contributory to good fire resistance. This is so, regardless of the fact that the aggregate seems to be the key. An aggregate which exhibits a low coefficient of thermal expansion, within the temperature range under consideration, would likely have favor over aggregates with a large coefficient; aggravated cracking and high internal stresses are otherwise induced in and around pieces of aggregate subjected to thermal shock. Thermal conductivity as well as heat or thermal capacity of aggregate also play an important role with reference to heat absorption and heat dissipation.

While little or no information is given in ASTM Descriptive Nomenclature of Constituents of Natural Mineral Aggregates (C 294), on the physical properties of aggregate which pertain to their fire resistant qualities, this standard is useful in pointing out those aggregates which contain silica and those which are primarily carbonates or calcareous in nature. In an ASTM symposium article [*7*], Ingberg catalogs aggregates into four general groups based on their fire resistance. Group I, calcareous aggregates, encompasses limestone and dolomite such as compose many crushed stone aggregates or as may occur as constituents of natural stone or gravel. Group I is said to give the least disruptive effects and the lowest temperature transmission, the latter being retarded by the calcination of the limestone and dolomite, which change, uses up some 430 cal/g for calcium carbonate and 340 for magnesium carbonate. Group II aggregates, the nonquartzose silicate aggregates composed mainly of feldspars, olivine, and amphiboles and pyroxenes such as hornblende and augite do not evidence cracking or spalling in fire resistance tests; these aggregates include many common rocks such as basalt and diabase (traprock), dolerite, andesite, gabbro, and syenite. In addition, pumice and artifical aggregates such as broken brick, expanded clays and shales, blast-furnace slag and the harder varieties of furnace clinkers or cinders likely lie in this group with fusion points in the range of 2100 to 2300 F. Group III aggregates which evidence cracking and spalling in fire tests comprise quartzose igneous rocks that are higher in silica content than Groups I and II. Granites, which are composed of quartz, feldspars, and ferromagnesian materials, also lie in Group III, along with sandstone, quartzite, and quartz-bearing schists and gneisses. Group IV comprises rocks consisting mainly of silica not combined to form silicates, and include quartzite, quartz, chert, and flint which cause cracking, shattering, and disruption of concrete at depths of 1 in. and more below the surface.

Aggregates high in silica appear to have a higher coefficient of thermal expansion than do those of low silica content [*8*]. This is brought out by the following listing published by the Bureau of Reclamation [*9*].

Rock	Coefficient of Thermal Expansion 10^{-6} per deg F
Basalt and gabbros	3.0
Marbles	3.9
Limestones	4.4
Granites and rhyolites	4.4
Sandstones	5.6
Quartzites	6.1

The coefficient of thermal expansion changes with increase in temperature up to 1000 F or higher. Likewise, degradation and change in crystalline structure of aggregate can occur at critical temperature, as evidenced by the sudden expansion which quartz exhibits at 1063 F [*9*].

Lightweight aggregates appear to have particular merit with regard to fire resistance. Many of these are the products of high-temperature furnaces or kilns and thus, after cooling, they usually have good stability during reheating which is probably due to their porous structure and glassy composition. Their capacity to retain moisture in concrete has been demonstrated to be beneficial in dissipation of heat, although in some instances too much retained moisture in conjunction with a tight void structure has been found detrimental as may also be the case with normal aggregate concrete. Lightweight aggregates include expanded perlite, exfoliated vermiculite, pumice, scoria, expanded slag, and the expanded clays, shales, and slates.

HIGH-TEMPERATURE RESISTANCE

Definition:

In evaluating the hardened concrete for its resistance to high temperature, consideration must first to be given to the meaning and intent of the terms "resistance" and "high temperature." Fire resistance *per se* is defined by ASTM Tentative Definitions of Terms Relating to Fire Tests of Building Construction and Materials (E 176 – 64 T) as follows:

> The property of a material or assembly to withstand fire or to give protection from it. As applied to elements of buildings, it is characterized by the ability to confine a fire or to continue to perform a structural function, or both.

ASTM Methods of Fire Tests of Building Construction and Materials (E 119) relates only to fire resistance and elements of structures. Essentially, ASTM Methods E 119, is the one and only established test in this category. However, this lone criterion for resistance to high temperature does not eliminate consideration of special empirical or arbitrary tests or both developed to simulate blast and heat generated by missiles or jet [*10*] airplane engines, or the heat to which a floor is subjected in a foundry either by radiation or by contact of hot coils or ingots being stored on the floor. Further, the introduction and use of solid propellants introduces a whole new field of concern wherein abrasion by solid particles accompanies terrific blast and heat. The use of the oxygen lance or other similar related fuel-powered devices for cutting through concrete is in effect a negative approach toward evaluating resistance of this material to high temperature.

Deterioration that results from these exposures is often cataloged arbitrarily, this classification being dependent upon how much damage can be tolerated. A loss-of-strength criterion of 80 per cent in one instance might be contrasted to "minute crazing will be permitted" in another.

FIRE-RESISTANCE TESTING

ASTM Methods E 119:

ASTM Methods E 119 may be used to evaluate the fire-resistance of walls, columns, floors, and other structural members. Any structural instability of a member, or its inability to prevent undue passage or spread of flame or heat, is considered a menace to the public or to adjacent structures while such a fire is underway. This method of test is not designed to evaluate the structural adequacy of any element with reference to the continuing use or occupancy of a building after the fire is out and the structure has cooled. Furnace temperatures in Methods E 119 are fixed by an established time-temperature curve which encompasses a stipulated 1000 F (538 C) at 5 min, 1700 F (927 C) at 1 hr, and 2300 F (1260 C) which must be reached at 8 hr unless, of course, the element has reached an end point before that time. ASTM Methods E 119 also places limits on the peak temperature of the unexposed surface, the passage of flame or gases through the element, and by option, its resistance to the cooling effects of a hose stream.

Some revision to Methods E 119 has recently been indicated, the most serious being based on the fact that the moisture content of a building element does have a marked influence on its fire resistance rating. One study reported by Harmathy [*11*] indicates that high-moisture content may result in spalling, but that if spalling does not occur, the presence of moisture is beneficial toward attainment of a high rating. Spalling in too moist a concrete may be caused by too rapid a buildup of steam pressure at some point below the surface. On the other hand, dry concrete or aggregate might spall just due to thermal stresses, the spalling tendency being increased with decrease in value of the factor:

$$\frac{\sigma_f(1 - \nu)}{E\alpha}$$

where:

σ_f = tensile strength,

ν = Poisson's ratio,

E = modulus of elasticity, and

α = coefficient of thermal expansion.

In this regard, the coefficient of thermal expansion of concrete lies somewhere in the range of that of the hardened cement paste, the sand, and the coarse aggregate. Ordinarily, the greater expansion of the cement paste is offset by the restraint offered by the coarse aggregate. The greater the amount of coarse aggregate in a given concrete, the greater is its influence.

FIRE RESISTANCE

A study by the Portland Cement Assn. [*12*] indicates that type of aggregate alone has a marked influence on the fire resistance of prestressed concrete beams. Lightweight aggregate of the expanded shale type was superior in this regard to a dolomitic normal weight aggregate which in turn had some advantage over a siliceous aggregate. At the exposed surface, calcination of the dolomite with its endothermic effect, retarded the advance of the high-temperature zone into the members; this caused some delay in the critical temperature rise in the prestressing steel. Companion studies also point out the influence of thickness of concrete cover over the prestressing steel, as well as that of the intensity of load carried by a prestressed beam.

Peter Smith in an Am. Concrete Inst. (ACI) article [*13*] on the effect of formwork or falsework fires on concrete (subtitled "New Concrete is Not for Burning") covers the effect of fire on new concrete as well as the repair and restoration of the concrete. His discussion is concerned with concrete at an age ranging from 1 hr to 40 days, and on the effect of fire on compressive strength, modulus of elasticity, spalling, cracking, volume change, and other properties of concrete and steel (reinforcing or structural) even to the point of considering prestressed concrete. Smith concludes that "new" concrete is not always irreparably damaged by fire and that it has shown remarkable resistance to such exposures even though it may not have attained much strength at the time of the incident.

Carlson [*14*], in an ACI symposium, reports on the effect of moisture, the size of specimen, conditions of restraint, surface temperature criteria, the effect of radiant heat, and of the comparison between tests in the United States and other countries. Benjamin [*15*] reports on fire tests of reinforced concrete as such and gives consideration to the following: (1) thermal factors (type of aggregate, moisture effect); (2) structural factors (concrete strength, and mix, cover, spalling, lateral restraint); (3) fire endurance ratings; and (4) plaster protection. Sheridan [*16*] discusses concrete as a protection material for steel sections, whereas Troxell [*17*] discusses prestressed reinforced concrete units. All four of these papers represent a comprehensive literature search into their particular fields of interest covering not only data and findings from the United States but also from other countries.

An extensive study made on concrete slabs *per se* was that by Menzel [*18*]. Horizontally cast slabs, 5 by 5 ft in the horizontal plane and 4, 6, or 8 in. thick, were tested in a vertical position and were subjected to standard fire-resistance curve temperatures. Highly siliceous sand and gravel, calcareous sand and gravel, and fine and coarse crushed Haydite were used as aggregates. The following effects of the fire tests were noted:

1. *Bond Between Aggregate and Cement Paste*—Dehydration, expansion and changes in the physical properties of the aggregate caused a reduction in strength of the concrete and of the bond of aggregate with cement.
2. *Spalling and Shattering*—No spalling was noted to have occurred although it was also established that the concretes were dry when tested. Shattering of coarse aggregate (siliceous gravel) particles occurred to depths of 1 to 1.5 in. (this loosened material would be easily removed by blast).
3. *Fusion*—After a period of $6\frac{1}{2}$ hr and possibly when the temperatures had reached about 2200 F, fusion occurred over portions of the exposed face of one of the slabs of expanded shale aggregate; this one slab performed satisfactorily for 9 hr at which time the test was terminated.
4. *Change of Shape*—The slabs were not unduly restrained and therefore warped upon heating.
5. *Cracking*—Many cracks appeared, the number and width increasing with increased thickness of slab. In general, cracks occurred on the "cool" side during fire exposure; when heating was discontinued and the exposed side was permitted to cool, it also began to crack.

The type of failure described above

under Item I of Menzel's study can be insidious in nature, since its effects are not readily visible. It is the loss of adhesion in the matrix of the concrete caused by the dehydration of the cement upon heating. This loss is a function of temperature and time and may not be visible until it is too late. The subsequent crumbling or breaking away of the surface may create a hazard, particularly when blast accompanies the heat.

Tests have also been made with regard to the effect of high temperature on the compressive strength of concrete. One such study reported by Malhotra [*19*] involved the testing of 6 to 30-week old 5-in. cubes stored at 75 F and 55 to 60 per cent relative humidity. These were heated with and without restraint (in compression) and tested for strength at various increments of temperature up to 600 C (1048 F). Malhotra's conclusions were:

1. The effect of temperature on the crushing strength of concrete is independent of the water-cement ratio within the range normally used.
2. The aggregate-cement ratio has a significant effect on the strength of concrete exposed to high temperature, the proportional reduction being smaller for lean mixes than for rich mixes.
3. Concrete under compressive stress of the order of its design stress has a smaller proportional decrease in strength than if the stress were absent.
4. The residual strength of heated concrete shows still further reduction in strength on cooling, being approximately 20 per cent less than the corresponding hot strength in the temperature range 200 to 450 C (328 to 778 F) for 1:4½ and 1:6 mix concrete.

HIGH TEMPERATURE AND BLAST

Simulation and Testing:

The above touches upon only one facet of resistance of concrete to high temperature, namely, the ASTM Methods E 119 fire-resistance and to closely related testing. There are further problems however which involve other exposures, rates of temperature rise, and even higher temperatures. Evaluation in these instances would be greatly simplified if a time-temperature-blast curve could be established for each particular case. Such curves are difficult to delineate in the first place, and the correlation of a test of a small specimen with what happened in the field can be very poor. Consider the problem presented by the use of jet engines in aircraft [*10*]. Concrete is exposed to heat and blast in test cells, warmup aprons, taxiways, and usually on the ends of runways where take-offs and landings are made. The problem is complicated somewhat by the extremes of surface condition at the time of exposure; the concrete pavement can be wet or dry, hot or cold, new or aged, repeatedly exposed or seldom so. Further, the degree of heat and blast is determined by the type of engine or engines in the aircraft, the height and inclination of the jet, and the duration of the exposure [*10*]. Duration of exposure depends greatly upon the operations procedure employed by each pilot or flight group and the regulations of the particular field in question regarding warmup and taxiing.

From these general observations, several types of exposure may be tentatively chosen for laboratory or field evaluation of concrete for jet-blast-resistant pavements. Consideration must be given to data which indicate that the temperatures immediately at the end of the tailpipe of a jet engine are about 1250 F without an afterburner, and range from 2500 to 3500 F with an afterburner. The blast has been estimated to reach velocities of 3500 ft/sec. The temperatures and the blast are diminished somewhat by the time contact is made with the concrete pavement, and they are reduced considerably when the angle of the jet is flat. In some instances, however, it is believed that the height and angle would be such that the air temperature immediately in contact with the concrete would be about 2000 F.

As to missiles, the intensity of their expected blast and heat is indicated by the characteristics of a test apparatus [*20*] designed to evaluate ablatives in connection with the space program. This test apparatus is said to also simulate the discharge from missiles already developed or else contemplated for use in the future. The article mentions temperatures of 5000 F and of gas velocities of 8250 ft/sec. In practice, the missile lifts off the launch pad; thus, the time of exposure is but a few seconds. Also, concrete in the blast area is usually deemed expendable, and one thus gives consideration toward refurbishing certain areas between launchings. Concern then centers around two factors, namely, feasibility of refurbishing between launchings and how costly will it be.

SUMMARY

Considerable research has been conducted with regard to the resistance of concrete to fire in accordance with ASTM Methods E 119 and to temperatures closely related to the standard fire curve. Also, isolated studies have been made on the effect of more severe exposures involving a more rapid temperature rise, some degree of blast, and higher temperatures. Concrete properly designed, placed, and cured performs markedly well in resisting fire, heat, and blast. Of greatest importance is the quality of the coarse aggregate although even here, only the degree of resistance is essentially affected. Nevertheless, specifications covering aggregates for use in concrete to be subjected to high temperatures would be improved by inclusion of limitations on composition and thermal properties. Adequate resistance is still obtained by conditioning (control of ambient temperature and humidity) the concrete after curing and by the choice of proper thickness of member or of cover over reinforcement.

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Hardened Concrete

RADIATION EFFECTS AND SHIELDING

BY MILOS POLIVKA,[1] Personal Member, ASTM

Portland cement concrete is an ideal material for use in construction of radiation shields. Although there are other materials that could be employed for radiation shielding purposes, concrete is not only economical, but it also has the advantage of being a material that can be cast into any desired homogeneous structural shape. Concrete is now commonly used for shielding of atomic research facilities, nuclear power plants, and for radiation medical and research units or equipment.

Conventional concrete of sufficient thickness can be and is being used for such purposes. However, where usable space is a major consideration, the reduction in the thickness of the shield is accomplished by the use of high-density concretes. Such high-density concretes, produced by the use of heavy aggregates, will usually have a unit weight ranging from 210 to 240 lb/ft^3, which is about 50 per cent higher than the unit weight of conventional concrete. They can, however, be produced with densities up to about 330 lb/ft^3 using iron as both fine and coarse aggregate. For certain types of biological shields there is a need to include materials of low atomic weight in the concrete.

At present there are no ASTM test methods or specifications for concrete or concrete-making materials written specifically for biological shield applications. However, several of the existing ASTM methods and specifications pertaining to concrete and concrete-making materials are applicable to shielding concretes. Specific references to some of these methods and to their applicability are covered later herein.

Extensive literature is available on the subject of concrete for radiation shielding. Pertinent papers were reviewed and are cited in the list of references. Recognition by Committee C-9 of the significance of concrete for radiation shielding as an engineering material is evidenced by the activities of its Subcommittee II-h on Concrete for Radiation Shielding.

This research subcommittee was established for the purpose of carrying out a study and research on the general problem of concrete for radiation shielding. After a three-year study, the committee acquired information which will be made available to the profession in the form of a symposium to be published by ASTM. This committee is also responsible for the preparation, and revision as needed, of a specification for aggregates for shielding concrete and of a descriptive nomenclature of constituents of aggregates for shielding concrete.

This paper consists of a review and discussion of radiation shielding and of properties of concrete and concrete-making materials for shielding purposes and of their effects and significance. The work of other writers on the subject, when used, is appropriately referred to.

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TYPES OF RADIATION

There are two general classes of radiation that are considered in the design of biological shields: (*a*) electromagnetic waves and (*b*) nuclear particles [*1*].[2]

Electromagnetic Waves:

Of the electromagnetic waves, the high-energy, high-frequency waves known as X- and gamma rays are the only types which require shielding for protection of personnel. Gamma rays are identical with X-rays except for the source. Gamma rays are given off spontaneously from naturally occurring heavy elements or from lighter elements produced in atomic piles or by accelerators [*2*]. Both X-rays and gamma rays have a high power of penetration but can be adequately absorbed by an appropriate thickness of concrete shield.

Nuclear Particles:

Nuclear particles consisting of nuclei of atoms or fragments thereof, include neutrons, protons, and alpha and beta particles. Of these, all but the neutrons possess an electric charge.

Neutrons, the heavy particles of atomic nuclei, do not carry an electrical charge and thus are unaffected by electrical fields surrounding atoms. They are slowed down only on collision with atomic nuclei. Neutrons have high powers of penetration but do not follow a defined path through a given material. Neutrons are classified as fast, intermediate (epithermal), and slow (thermal) neutrons. The energy range of fast neutrons is between 10,000 and 10,000,000 ev, of intermediate neutrons between 0.5 and 10,000 ev, and of thermal neutrons less than 0.5 ev.

Protons and alpha and beta particles carry electrical charges which interact with the electrical field surrounding the atoms of the shielding material, thus decreasing the energy of the particle. They generally do not constitute a separate shielding problem, although accelerated protons at high-energy levels may require heavy shielding comparable to that required for neutrons of equal energies.

Tolerance Levels:

Concrete shields are installed to protect personnel from radiation emitted by nuclear reactors, accelerators, nuclear explosions, and some medical and research units or equipment. The magnitudes of radiation must be determined, using theoretical calculations and experimental data, to properly design a biological shield that will insure that the level of radiation on the outside of the shield is maintained below the maximum permissible intensity. Information on the tolerance levels for gamma rays and neutrons was published by the National Bureau of Standards [*3*,*4*].

Low Background Concrete:

In addition to the use of concrete shields as protection from radiation, there is also the problem of designing shields which will minimize the so-called "background" radioactivity. As reported by Wollenberg and Smith [*5*] concrete was successfully used as a shield against radioactivity in structures enclosing areas where low levels of gamma radioactivity are measured, that is, low-background counting facilities. Such concretes must not only provide a shield from extraneous radioactivity, but the concrete itself must be low in radioactivity.

SHIELDING PROBLEMS

As discussed above, the principal types of radiation requiring consideration in the design of a shield are gamma rays and neutrons. A shield adequate to

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

reduce gamma radiation is normally also sufficient for protons and alpha and beta particles. Thus, the general shielding problem resolves itself into attenuation of neutrons and gammas. Almost any material of sufficient thickness can serve satisfactorily as a shield, but neutrons and gammas are not equally attenuated by the same materials.

Neutron and Gamma Ray Shielding:

Neutron Shielding—For an efficient neutron shield it is often desirable to incorporate three classes of materials. The shield should contain some heavy material, such as iron, whose atomic mass is 56, or elements of higher atomic number. These heavy elements slow down the fast neutrons by inelastic collisions. It is desirable to have light elements, preferably hydrogen, to further slow down the moderately fast neutrons through elastic collisions. Hydrogen is particularly effective because it weighs about the same as a neutron. Finally, it is necessary to remove the slow, thermal neutrons by absorption. Hydrogen is effective in this action, but in the process emits a 2.2 million volt gamma ray, which requires considerable shielding itself. Boron, on the other hand, has not only a very high cross section (absorbing power) for neutrons, but emits gamma rays of only 0.478 million volts. For these reasons boron-containing materials are often used in neutron shields. The gamma rays given off during absorption of neutrons are a factor in the required shield thickness.

Gamma Ray Shielding—Gamma radiation may originate inside the shield through neutron capture as well as from the primary source behind the shield. Attenuation of only the high-energy gamma radiation (1 to 10,000,000 v) is here discussed but not that of the lower energy X-rays. Most materials attenuate gamma radiation primarily by the Compton scattering process [*2*] with the attenuation being approximately proportional to the mass of the material in the radiation path, but very little influenced by the type of material, that is, different materials of the same mass have approximately equal protective properties against gamma radiation [*6*].

Shielding Requirements:

Concrete shields are usually of two distinct types: (*a*) those required to attenuate gamma radiation only and (*b*) those required to attenuate fast neutrons and associated gamma radiation [*7*]. For the first case, high density and low cost are of prime importance. For the second case, it is desirable to have materials of high or medium atomic mass (to slow down fast neutrons); materials of low atomic weight, especially hydrogen (for reducing neutrons of intermediate range to the thermal range); and finally materials for absorbing thermal neutrons. These requirements for a neutron shield are summarized in more detail by Henrie [*8*].

Shielding Ability of Concrete:

Concrete is an excellent shielding material that possesses the needed characteristics for both neutron and gamma ray attenuation, has satisfactory mechanical properties, and has a relatively low initial as well as maintenance cost. Also the ease of construction makes concrete an especially suitable material for radiation shielding.

Since concrete is a mixture of hydrogen and other light nuclei, and nuclei of higher atomic number, and can be produced within a relatively wide range of density, it is efficient in absorbing gammas, slowing down fast neutrons, and absorbing resonance and slow neutrons [*9*]. The hydrogen and oxygen, contained in chemically combined form in the hydrated cement, moderate the neutron flux satisfactorily. The oxygen may also be present in the concrete in another form in addition to water; concrete with silica aggregate is about one-half oxygen.

Intensity of gamma radiation rather than of neutrons often determines the thickness of the shield, in which case the required thickness will vary almost directly with the density. The efficiency of a concrete shield of given thickness depends mainly on its density and uniformity. The density of concrete can be controlled within wide limits by selection of appropriate heavy aggregates. The desired uniformity of the shielding quality requires that the concrete form a homogeneous mass with uniform distribution of the aggregate particles. Excessive settling, segregation, or formation of pockets during placing may cause excessive radiation to penetrate such faulty sections.

TABLE 1—AGGREGATES USED IN SHIELDING CONCRETE.

Natural Mineral		Manufactured	
Local sand and gravel		Crushed aggregates	
Calcareous Siliceous	(2.5 to 2.7)	Heavy slags	(~5.0)
Basaltic	(2.7 to 3.1)	Ferrophosphorus	(5.8 to 6.3)
Hydrous ore		Ferrosilicon	(6.5 to 7.0)
Bauxite	(1.8 to 2.3) [15 to 25%]	Metallic iron products	
Serpentine	(2.4 to 2.6) [10 to 13%]	Sheared bars Steel punching	(7.7 to 7.8)
Goethite Limonite	(3.4 to 3.8) [8 to 12%]	Iron shot	(7.5 to 7.6)
Heavy ore		Boron additives	
Barite	(4.0 to 4.4)	Boron frit	(2.4 to 2.6)
Magnetitite Ilmenite Hematite	(4.2 to 4.8)	Ferroboron	(5.0)
Boron additives		Borated diatomaceous earth	(~1.0)
Calcium borates		Boron carbide	(2.5 to 2.6)
Borocalcite Colemanite	(2.3 to 2.4)		
Gerstley borate	(2.0)		

NOTE—Specific gravity is shown in ( ).
Water of hydration is indicated in [ ].

Materials used in a shield should not be subject to decomposition or weakening under the influence of radiation. Mather [9] reports that data reviewed indicate that the radiation stability of concrete is adequate for most applications. In some cases, such as removable shielding plugs, materials that become highly reactive when exposed to intense thermal neutron radiation should be avoided. Troublesome elements include vanadium, chromium, manganese, cobalt, and others [7]. Also concrete containing potentially deleteriously reactive cement-aggregate combinations should be avoided.

AGGREGATES FOR SHIELDING CONCRETE

Concrete shields may be small structures housing a research nuclear unit or may be massive monolithic structures or structures consisting of precast assembled elements of various sizes. The thickness of the concrete shield will vary depending on the type of nuclear unit but may range up to 6 or 8 ft. Conventional concrete has been used to provide radiation shielding. If there are no space limitations, conventional concrete will provide a most economical shield.

Frequently, however, it is desired to produce concrete of improved properties with respect to shielding ability, especially when space limitations require that the thickness of the shield be reduced. In such cases the shielding ability of the concrete is increased by increasing its density and in the case of neutron shields by including a sufficient amount of light elements. The increase in the density of the concrete is accomplished by the use of heavy aggregates, such as naturally occuring iron ores, manufactured iron and steel, ferrophosphorus, and barite. Aggregates containing bound water are sometimes added to the mix to shield against fast neutrons. They include hydrous-iron ores, serpentine or bauxite. Also, to shield against neutrons, boron-containing admixtures are sometimes added to the concrete.

Information on conventional concrete and concrete-making materials is thoroughly covered by the various papers of this publication. The remainder of this paper will refer to concretes whose properties were enhanced with respect to their shielding ability.

Types:

Naturally occurring iron ores, manufactured iron and steel, ferrophosphorus, and barite are the major heavy aggregates that have been used to increase the density of concrete. A listing of these aggregates as prepared by Davis [7,*10*] is given in Table 1. Included in this table are boron-containing additives used in neutron shields.

Natural mineral aggregates can produce concrete densities up to 240 lb/ft^3. The most commonly used have been barite, magnetite, and ilmenite. Along with hematite, these are used chiefly for gamma attenuation. To improve neutron attenuation, hydrous ores including goethite, limonite, serpentine, and bauxite, and borates such as colemanite, boron frit, and borocalcite have been used. The manufactured heavy aggregates listed in Table 1 (except for the boron additives) are only used when the required concrete density is greater than about 250 lb/ft^3 [*10*].

Composition:

In general, the exact chemical composition of heavy aggregates is not critical as long as the required density is met. As a control of the uniformity of the high density of some of these aggregates chemical limitations may be applied. For example, barite ore should have a barium sulfate content greater than 90 per cent, while the iron content of magnetite and limonite aggregate should be in excess of 60 and 55 per cent, respectively. Such limits, however, will greatly depend on the economy of using easily available materials which may justify using lower values. For example, magnetite ore from Wyoming has an iron content of about 50 per cent, while ilmenite ore from Quebec has an iron content of only 38 per cent. The specific gravity of both of these aggregates is in excess of 4.5. To require the iron content to be 55 per cent or greater would eliminate these two reliable sources of heavy aggregate [*10*].

Composition of aggregates becomes very important when they are to be used in low-background counting facilities requiring aggregates of low levels of natural gamma activity. Of primary concern are the concentrations of potassium, uranium, and thorium. Based on extensive studies, Wollenberg and Smith [*5*] indicate that serpentinized ultramafic rock is the most suitable source of low-background aggregate.

Radiation Attenuation and Absorption:

The capacity of the various heavy aggregates to absorb gamma rays is almost directly proportional to their density. Also the heavier elements are more effective in absorbing fast neutrons by inelastic collisions than are the lighter ones. Davis [*7,10*] gives representative values of absorption factors for various aggregates and other concrete-making materials.

However, density is not the only factor to consider in the selection of an aggregate for a neutron concrete shield. The desired increase in hydrogen content, required to slow down fast neutrons, can be accomplished by the use of hydrous ores. These materials contain a high percentage of water of hydration (fixed water content) as shown in Table 1. However, on heating of the concrete some of this fixed water in the aggregate may be lost [*10*]. Limonite and goethite are reliable sources of hydrogen as long as shield temperatures do not exceed 390 F (200 C), whereas serpentine is good up to at least 750 F (400 C).

Measurement and Significance of Properties:

Members of Subcommittee II-h of ASTM Committee C-9 have made an evaluation of ASTM standards on aggregates with regard to their applicability to heavy aggregates. Most of the pertinent existing standards are applicable in their present form. A discussion follows of some of the important properties and their significance.

Nomenclature and Mineralogy—ASTM Descriptive Nomenclature of Constituents of Natural Aggregates (C 294) does not include a number of the more probable constituents of heavy aggregates, and treats several of the important constituents of heavy aggregates, such as iron oxides, only in their capacity as accessory constituents of rocks in use as normal aggregates. Subcommittee II-h of ASTM Committee C-9 has chosen to prepare a new document rather than request revision of Standard C 294. Such a descriptive nomenclature of constituents of aggregates for shielding concrete is being prepared by the Subcommittee.

ASTM Recommended Practice for Petrographic Examination of Aggregates for Concrete (C 295) applies to aggregates for shielding concrete as well as it does to other aggregates. Mather [*11*] states:

> The high-density aggregates, and aggregates containing water that are used in shielding concrete, present special problems, but they are still problems in which petrographic identification of reactive substances, or substances likely to produce problems in concrete production and placement, can be useful.

Chemical Properties—Raphael [*12*] has reported expansions of mortar bars, tested according to ASTM Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar Bar Method) (C 227), containing barite aggregate and high-alkali Type I cement, which were large enough to cause the combination to be regarded as alkali reactive. These expansions might have been caused by reactive silica or by calcium sulfate in the aggregate.

Method C 227 is applicable to heavy aggregates. Based on results of a limited study made by W. L. Dolch, for the information of Subcommittee II-h, it appears that ASTM Test for Potential Reactivity of Aggregates (Chemical Method) (C 289) is also applicable to heavy aggregates.

The origin of the majority of deposits of heavy aggregates makes the presence of organic impurities very unlikely. However, presence of such organic impurities should be checked for by the use of ASTM Test for Organic Impurities in Sands for Concrete (C 40). If such a test is included in a specification for heavy aggregate it should contain an explanation similar to that given for lightweight aggregates, ASTM Specification for Lightweight Aggregates for Structural Concrete (C 330 – 64 T), as follows:

> Heavy aggregates that, upon being subjected to the test for organic impurities, produce a color darker than the standard shall be rejected, unless it can be demonstrated that the discoloration is due to small quantities of materials not harmful to the concrete.

Deleterious Constituents—ASTM tests for deleterious constituents of aggregates include tests for clay lumps (ASTM Test for Clay Lumps in Natural Aggregates (C 142 – 64 T)) and lightweight pieces (ASTM Test for Lightweight Pieces in Aggregates (C 123)) are applicable to, and can be used in specifications for, heavy aggregates. The test for hardness (ASTM Test for Scratch Hardness of Coarse Aggregate Particles (C 235 – 62 T)), however, when applied to heavy aggregates, needs some interpretation.

The hardness of the minerals magnetite, hematite, and ilmenite, which produce ores of high specific gravity (4.2 to 4.8) is about 5 to 6 on Mohs' scale. Barite ore, which has a specific gravity of 4.0 to 4.5, has a Mohs' hardness (of the mineral) of only 2.5 to 3.5. The Mohs' hardness of calcite, by definition, is 3. Thus if Method C 235 is intended to classify calcite as just barely harder than "soft," it would be expected to classify barite sometimes as "hard" and sometimes as "soft." Therefore, it would not be reasonable to ask for a barite aggregate under a specification that includes a limit on the amount of soft particles, as is the case of ASTM Specification for Concrete Aggregates (C 33), for particles scratched by the C 235 test. For these reasons, it is necessary to use careful judgment in the application of Method C 235 to heavy aggregates.

Gradation—The gradation requirements of heavy aggregates can be within the limits given in Specification C 33. The sample of heavy aggregate used in the sieve analysis should be greater in weight than the samples specified in Sections 4(*b*) and (*c*) of ASTM Test for Sieve or Screen Analysis of Fine and Coarse Aggregates (C 136). This increase in weight should be obtained by multiplying the specified weight (Method C 136) by the ratio of the specific gravity of the heavy aggregate to that of ordinary aggregate (for which a value of 2.65 may be assumed).

Unit Weight—The unit weight of heavy aggregates can be determined by the procedures of ASTM Test for Unit Weight of Aggregate (C 29). However, it might be desirable to employ larger capacity measures than those specified in Section 2(*c*). For example, a 1-ft^3 capacity measure is much too small for 4-in. aggregate. Also, the use of a vibrating table in lieu of rodding (Section 5 of Method C 29) is suggested as a better procedure for heavy aggregates.

Specific Gravity—In the design of a concrete shield the unit weight selected for the concrete is of primary importance. The specific gravity of the aggregate is the major factor influencing unit weight and is, therefore, of considerable significance in specifications for heavy aggregates. The specific gravity, for both fine and coarse aggregate, is determined on a saturated surface-dry basis in accordance with appropriate sections of ASTM Test for Specific Gravity and Absorption of Fine and Coarse Aggregates (C 128 and C 127), respectively. Again, the sample weights should be increased according to the procedure described earlier under Gradation. To produce concrete weighing 230 to 240 lb/ft^3, the aggregate must have a specific gravity of 4.5 or greater. To produce concrete weighing around 300 lb/ft^3, the aggregate must have a specific gravity of 6.0 or greater.

It is necessary in specifications for heavy aggregate to give the minimum values of specific gravity. Davis [*10*] gives the following minimum values: limonite or goethite 3.25, magnetite 4.50, ferrophosphorus 6.10 for fine and 5.90 for coarse aggregate. The range of specific gravities of the various aggregates used in shielding concrete is given in Table 1.

Resistance to Abrasion—The resistance of heavy aggregates to abrasion and their tendency to fracture during handling and mixing varies considerably, and allowance must be made for the natural characteristics of the heavy ores being used [*10*]. Magnetite and ilmenite, which can be purchased in satisfactory gradings, are harder than barite and the hydrous ores, goethite and limonite, and are less prone to produce excess fines during handling and mixing [*9*]. Since concrete shields are generally not subjected to severe abrasion, aggregates which do not pass the abrasion resistance requirement of Specification C 33 (maximum abrasion loss of 50 per cent) may be used, provided these aggregates produce concrete of otherwise satisfactory properties.

CONCRETE FOR RADIATION SHIELDING

The special concretes used for the construction of shielding structures are distinguished from conventional concretes primarily by their higher density, obtained through the use of heavy aggregates, and in some cases by containing boron additives to improve attenuation of neutrons [*13*]. The practices to be employed in the proportioning, mixing, and curing of the shielding concretes are basically the same as those used for conventional concretes. Test methods used for the evaluation of the properties of fresh and hardened conventional concretes are discussed in this publication by various authors. In this paper only the special properties of shielding concretes, some of which are of less consequence in conventional concrete structures, are discussed.

Special Properties and Their Significance:

In the selection of mix proportions for a concrete to be used for shielding purposes several properties need special consideration. They include the workability and setting characteristics of the fresh concrete and the density and strength of the hardened concrete. The effects of high-temperature exposure and thermal cycling on the properties of hardened concrete need also to be considered.

Workability—To facilitate the proper placing of concrete in forms (frequently of complex shape and containing many embedded items) and to insure the integrity of the concrete in the shield (avoiding segregation and cold joints), the concrete mix must be extremely workable. Because heavy aggregate mixes tend to be harsh, more fine aggregate is usually used than in conventional concrete, and the sand frequently contains a high percentage (10 to 16 per cent) of material passing the No. 100 sieve [*7,10*]. Cement contents of the concrete are usually high (6 to 8 sacks per cubic yard and even higher), because of desired workability or fixed water content. Frequently, when a shield includes closely spaced reinforcement or a high concentration of embedded items, it becomes necessary to employ other than conventional methods of placing, such as the preplaced aggregate method.

Setting Characteristics—The setting characteristics of a concrete need to be considered when boron additives are used in neutron shields, since some of these borates may adversely affect the setting and strength properties of the concrete. As discussed earlier, boron is an efficient element in shielding against neutrons.

However, most naturally occurring borates like colemanite and borocalcite are somewhat soluble, and borate solutions are powerful retarders of the set of concrete and also of the rate of strength gain [*8*,*9*]. In addition, borate ores frequently contain impurities that either tend to reduce the ultimate strength of concrete or cause erratic setting behavior or both. Henri [*8*] discussed the influence of boron-containing additives, such as colemanite, borocalcite, and boron frit, on the setting characteristics of concrete. Colemanite is generally considered the poorest of the three in this regard However, it has the advantage of being more economical in some geographical areas. It contains about 10 per cent boron. Borocalcite is a calcium borate similar to colemanite and contains about 13 per cent boron. It also causes erratic setting characteristics of the concrete. To reduce the effect of these naturally occurring borates, all the fines passing a No. 30 sieve are usually eliminated. This lessens the exposed surface area for a given weight of the material and thereby reduces its solubility. These materials were successfully used in concretes, frequently in conjunction with calcium chloride (about 1 per cent by weight of cement) to overcome the retardation.

TABLE 2—DENSITIES OF SHIELDING CONCRETES COMMONLY SPECIFIED FOR GIVEN HEAVY AGGREGATES.

Type of Aggregate	Density, lb/ft^3
Barite	215 to 225
Magnetite	220 to 235
Ilmenite	220 to 240
Ferrophosphorus	285 to 300
Barite and boron additive	200 to 215
Magnetite and boron additive	210 to 225

Low-sodium boron frits are an excellent source of boron (about 18 per cent) and have little effect on the setting characteristics of concretes. These materials, being refined products produced by fusion of different compounds, are more costly than the naturally occurring borates.

It is highly desirable that prior to the use of borate additives a thorough evaluation of their effects on the properties of concrete, especially setting characteristics, be made.

Density—Though the density or unit weight of the concrete used in a shield is a major consideration, the designer also must consider all of the shielding characteristics of the concrete, as well as cost and construction problems. As stated earlier, conventional concrete (weighing say, 150 lb/ft^3) provides the most economical and is the most commonly used shielding material. Only due to limited space or other conditions of construction will concretes of higher densities be employed.

The density of the concrete is principally governed by the specific gravity of its aggregate, as previously discussed. The density is also influenced by the cement, water, and air content of the concrete mix. ASTM Test for Weight Per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138) is applicable to the determination of the unit weight of shielding concrete. Typical ranges of densities commonly specified for concretes made with some of the heavy aggregates are listed in Table 2.

Densities of about 330 lb/ft^3 can be produced when using iron as both fine and coarse aggregate. Densities other than those shown in Table 2 can be obtained by the use of various combinations of natural or manufactured aggregates. References included with this paper give the properties of many typical concrete mixes containing heavy aggregates.

Strength—Although structural strength may be a requirement for high-density shielding concrete, this property usually causes no problem because sections are thick and the mixes have high cement content [*14*]. For structural shielding concrete, 3000 to 5000 psi compressive strength is usually sufficient; for massive walls, 2000 psi is adequate. However, concrete for radiation shielding must have adequate strength to resist cracking during its service life. Some of the strength-determining properties of hardened concrete are discussed by Lott and Kesler [*15*].

In a recent study by the Corps of Engineers [*9,16*], it is shown that high-strength concrete can be made using materials that provide high density. The high-density, high-strength concrete produced for this investigation was made with a Type I portland cement, using magnetite or ilmenite aggregate of ½ in. or 1½ in. max size. The concretes had cement contents of 7.7 to 10.3 sacks per cubic yard, water-cement ratios of 0.30 and 0.35 by weight, and slumps of about ½ in. The unit weight of these concretes was about 230 lb/ft^3. The compressive strengths at 7 days were in the range 7600 to 9400 psi, at 28 days, 9000 to 11,000 psi, and at 90 days, 10,000 to 12,000 psi. It is concluded in this study [*9,16*], that although no new technology is needed to produce uniform high-density concrete of high strength, adherence to the best practices in production and control of such concretes is absolutely essential.

Effects of High-Temperature Exposure—The influence of high temperatures and thermal cycling on properties of concretes are of major importance in the design of concrete shielding structures [*17*]. These effects of temperature were studied by Davis [*7,13,17,18*], and the following evaluation of effects of temperature is taken from his most recent paper [*18*].

Fortunately, from a strength standpoint, concrete in a reactor shield is usually over six or nine months old before it is subjected to nuclear heating and is, therefore, mature in hydration and strength.

The loss in compressive strength that occurs when mature concrete is subjected to high temperatures ranges from 20 to 50 per cent, even when the concrete is heated as high as 600 or 800 F (320 or 430 C) for long periods. However, for important structural members, concrete should not be exposed continuously to temperatures much above 500 to 600 F (260 to 320 C). Concrete is almost completely dehydrated when heated above 800 F (430 C). If made with heat-resistant aggregates, it may still retain 25 to 50 per cent of its strength at temperatures as high as 1300 or 1400 F (700 or 760 C). On the other hand, if unstable aggregates or high water-cement ratios (over 0.60 by weight) are used in the mix, deterioration at temperatures above 600 to 800 F (320 to 430 C) may be appreciable.

If the concrete is subjected to wide and frequent fluctuations in temperature, the loss of strength of the concrete may be several times as great as that observed for concrete exposed to a constant high temperature.

If concrete contains special aggregates, such as limonite or serpentine, to provide hydrogen for neutron attenuation, the temperatures will be limited to a safe value below that which dehydrates the aggregate: about 350 to 400 F (180 to 200 C) in the case of limonite or as high as 700 F (370 C) for serpentine. On the other hand, the maximum temperature in a concrete shielding structure may be limited to less than 200 F (90 C) to minimize moisture loss, temperature gradients, or surface cracking.

Davis [*17*] shows that at high temperatures, tensile, flexural, and bond strength are affected more than compressive strength. In most shielding structures, thermal effects are of prime consideration in design.

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STP169A-EB/Apr. 1966

AIR CONTENT AND UNIT WEIGHT

By S. B. Helms[1]

Accuracy in the determination of unit weight of hardened concrete can be attained using relatively simple procedures based (*a*) on displacement methods, such as suspended-immersed weighings of samples on sensitive scales, or (*b*) on weight-in-air of the sample divided by its volume as calculated from dimension measurements. The latter procedure is especially adaptable for determinations on dry specimens using precise measurements of linear dimensions within an accuracy of one per cent. Apparatus and instruments for these purposes are familiar to all, and, in regard to matters of significance, elucidation of procedure details seems unnecessary.

On the other hand, any technique for determination of air content, after the concrete has set, must be more complex and would require specialized equipment if exact information is desired. Where an estimated air content will suffice to differentiate or to be indicative of the general level of air content, less discriminating techniques, predicated on precise density measurement, specific gravities, and known or determinable proportions of the ingredients, are available. While methods for unit weight are traditional, those for air content have developed since the time the advantages of entrained air became apparent, as the means for studying the effects of air content on other properties of the concrete.

Unit Weight of Principal Types of Concrete and Ranges of Air Content

To describe the various concrete types which are of commercial significance in one illustration, a chart known as the Unit Weight Block Diagram has been devised. Figure 1 serves to indicate the scope of this paper, insofar as air content and unit weight may be considered, whereas the other characteristics of concrete in certain major categories (that is, cellular, lightweight, radiation shielding) are covered separately, as topics of special interest in this publication.

Figure 1 is not intended to delineate exact limits, since it is well understood that important factors, such as moisture content and richness of mix, have an appreciable effect on the density of any given category. The projection of density of the fresh concrete to the hardened basis also involves changes related to bleeding (water gain) and settlement. Voids in vesicular aggregates contribute to reduced density but are not considered to be part of the air content charted on the figure. The arbitrary dividing lines between zones were drawn to correspond to available data in published references. Further, for purposes of this discussion, it seems essential to arbitrarily subdivide and classify concretes, according to their air contents, as: "non-AE" 0 to 2 per cent, "air entrained" 2 to 10 per cent, and "aerated" 10 to 30 per cent air. Other subclasses, according to unit weight, are: "lightweight" 20 to 120 lb,

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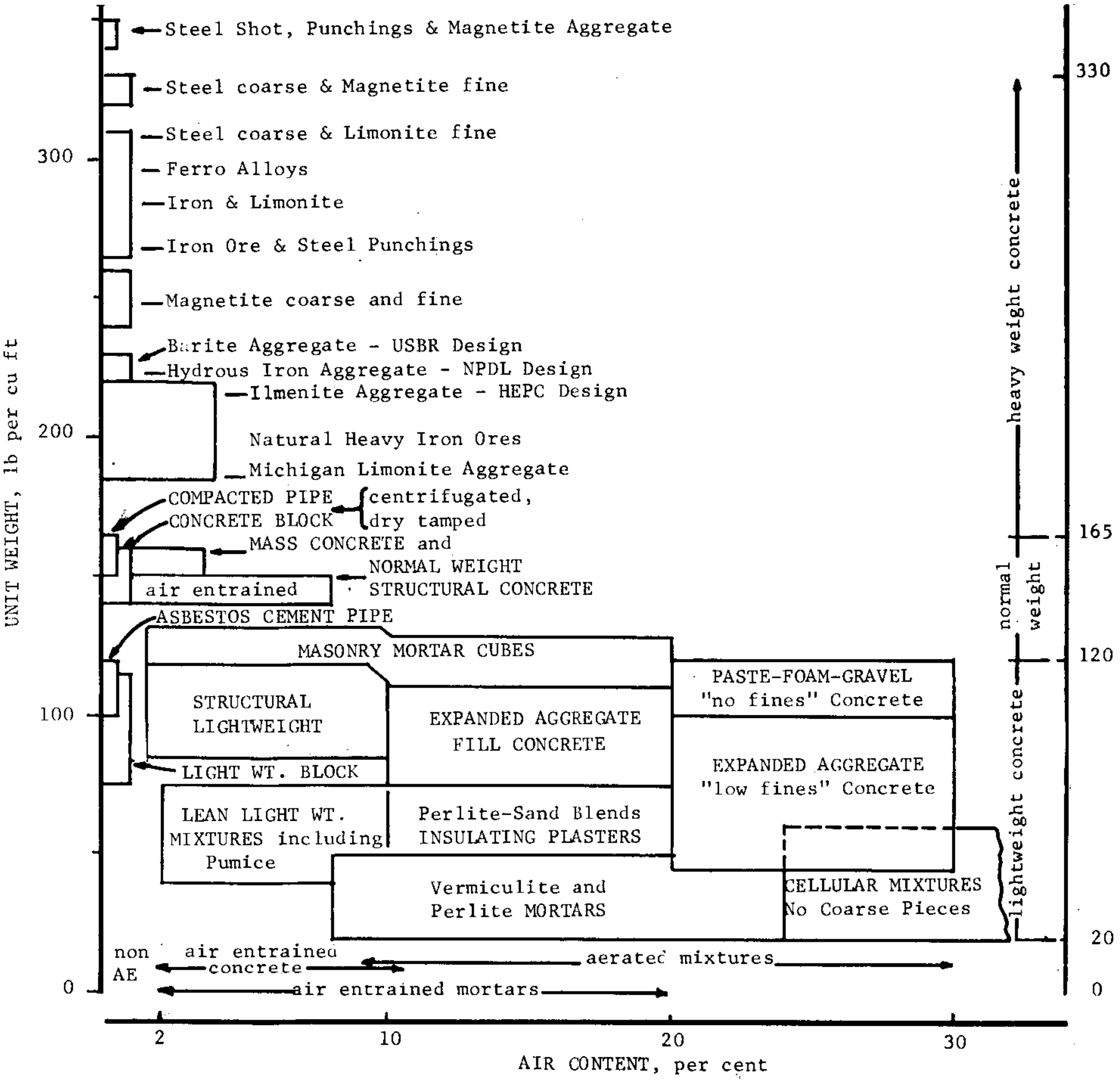
"normal weight" 120 to 165 lb, and "heavyweight" 165 to 330 lb/ft^3. Moisture content and some other factors having a minor effect on density were not considered in constructing the block diagram because of its descriptive nature.



NOTE—USBR = U.S. Bureau of Reclamation, NPDL = North Pacific Division Laboratory Corps of Engrs, and HEPC = Hydro-Electric Power Commission of Ontario.

FIG. 1—Unit weight block diagram.

ABRIDGED REVIEW OF DENSITY PRINCIPLES

In Feret's studies [*1*],[2] the "density" of mortar was represented by the total volume of the solid particles per unit volume. This could be termed the first application of the method of absolute volumes, which is involved both in proportioning and in the analysis of the physical constants of concrete. Feret assigned letters (*c* for cement, *s* for sand, *e* for water, *v* for air voids) to represent the absolute volumes of mortar constituents. The term density ordinarily employed is on the basis of weight per unit volume. Detailed discussions of the validity of relationships established by Feret are given in Refs. [*2* and *4*].

Using Feret's letter designations, it will be noted that the term $c/(1 - s)$ is a cement-space ratio, whereas $c/[1 - (c + s)]$ is the cement-voids ratio $c/(e + v)$. After the relationship between water content and richness of mix was established by Abrams' water-cement ratio, Talbot and Richart [*5*] developed relationships of strength and voids and introduced the concept of the cement-space ratio which now may be applied to results of air entrained and lightweight concrete. Figure 12 of the Talbot and Richart report on Series 2-G mortars and concretes [*5*], made with Wabash River aggregates, shows a linear relationship between unit weight of concrete and the voids in concrete represented by the line:

Unit weight, lb per cubic ft

$$= 167\ (1 - v - c) + 193\ c + 55\ v$$

This closely fitted the experimental points for these materials. Here v and c are absolute volumes of voids and cement, v being the sum of water and air voids. Talbot and Richart recognized that levels of water content affected compressive strength at a given ratio and employed a strength reduction curve to refine their charts of compressive strength versus cement-space ratio.

The reader is reminded that unit weight of concrete is less sensitive to water-cement ratio than to voids-cement ratio; thus, changes of unit weight will be found to be indicative of air content of air-entrained concrete. Incidentally, Feret [*6*] distinguished between "air-entrained" in mortar, water voids, and cavities quite a long time ago. This consideration has significance since no measurement of total air in fresh concrete will discriminate the different types and sizes of air voids, and microscopic examination of the interior structure of hardened concrete is required for this classification. There is one example of this (Fig. 2) which will be mentioned later.

TABLE 1—DATA FROM MASS CONCRETE INVESTIGATIONS.[a]
Unit Weight and Strength of Hand Rodded and Vibrated Specimens

Aggregate Maximum Size	Proportions by Weight	Slump, in.	Paste[b] Vibrated, volume %	Unit Weight, lb/ft³		Compressive Strength, 7 days, psi	
				Rodded	Vibrated	Rodded	Vibrated
No. 4	1:2.98	3.6	42.9	137.2	137.7	2360	2400
⅜ in.	1:2.80:1.50	3.8	36.7	141.8	140.6	2480	2590
¾ in.	1:2.79:2.65	3	30.1	148.0	149.2	2450	2480
1½ in.	1:2.46:4.26	3.4	26.5	152.9	155.1	2440	2400
3 in.	1:2.34:6.07	3.2	22.4	154.6	158.1	2280	2140

[a] Condensed from Table 61, Bureau of Reclamation, *Bulletin 4*, p. 154, Part VII, "Cement and Concrete Investigations, Boulder Canyon Project."
[b] Water-cement ratio 0.56 by weight.

The principles advanced by Talbot and Richart (cement-space ratio concept) were utilized by Morris [*7*] who developed a mortar voids method for designing concretes. Morris' paper contains several interesting illustrations which correlate strength with the cement-space ratio as shown in the original Bulletin No. 137 [*5*]. The concepts of Talbot and Richart were extended to the design of air entrained concrete mixtures by Thornburn [*8*]. Charts of the type used by Morris have unique utility in establishing strength versus voids correlations in presentation of design data for the lightweight insulating or aerated concrete classes described by Fig. 1.

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

DATA SHOWING EFFECT OF AMOUNT OF PASTE AND DEGREE OF COMPACTION

Since pastes in the practical range of 5 to 9 gal of water per sack of cement have an air-free unit weight in the range of 100 to 120 lb/ft^3, whereas densities of normal aggregates usually range from about 160 to 175 lb/ft^3,[3] it is clear that a reduction of paste volume by substitution of aggregate will increase the unit weight.[4] Table 1, applying to mass concrete, is quite interesting as an example of increased unit weight due to reduction of the amount of paste or mortar in the mixture. In mixtures of stiffer consistency, 2 in. slump or less, the degree of compaction as effected by vibration would have a more pronounced effect.

Important factors in the consolidation of concrete are reviewed in the report of ACI Committee 609.[5] By using vibration to facilitate compaction it is practical to lower the sand proportion and reduce the slump required for proper placing. Each of these changes permits lower water content and thereby increases unit weight. Improved densification combined with such adjustments of mix proportions also means that for a given cement content the mortar or paste will be of better quality (less water). The advantage of proper compaction of concrete in the field is as obvious as the disadvantages of incomplete or improper consolidation.

The committee report cites further benefits from vibration where revibration of a previous lift aids the rearrangement of aggregate particles and the elimination of entrapped water under aggregate and steel reinforcement, as well as eliminating extraneous air voids, with the result that bond is improved. Published results of effects of vibration have shown[5] that overvibration can reduce entrained air to low levels. However, the committee reports that extra vibration to eliminate undesired large voids "will not damage the parameters of the air void system . . . essential to best durability" if the concrete initially contains the amount of air recommended in ACI 613-54. While half of the initial air could be lost due to overvibration, the requirements for frost resistance apparently would still be met unless the *initial* air content was lower than that prescribed in the ACI 613 recommended practice.

RECOGNITION OF ADVANTAGES OF ENTRAINED AIR

A study [*9*] launched about 1935 showed that cores from certain experimental pavements having high resistance to freezing and thawing contained portland cement that had been blended with either of two natural cements which happened to contain about 0.07 per cent of fat or grease. Lowered unit weight was the clue leading to understanding that resistant slabs contained larger amounts of entrained air than did nonresistant slabs, and studies of air-entraining materials were started. It has been stated [*10*] that the first work correlating freezing-and-thawing tests with air content was reported by the Portland Cement Assn. in June, 1938. But the term "air-entraining cement" was not introduced until after their Study of Cement Performance was instituted in 1941, when these were known as "treated" cements. Improved scaling resistance of pavements made with-"treated" cement was attributed to entrained air by Moore [*11*] who stated:

> Both Vinsol resin and tallow reduce the unit weight of concrete by introducing air,

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[3] Specific gravity as measured by ASTM Test for Specific Gravity and Absorption of Coarse and Fine Aggregates (C 127 or C 128).

[4] Measured by ASTM Test for Weight Per Cubic Foot, Yield, and Air Content of Concrete (C 138).

[5] ACI Committee 609, "Consolidation of Concrete," *Journal*, Am. Concrete Inst., April, 1960; *Proceedings*, Vol. 56, p. 985.

uniformly distributed throughout the mass in microscopic voids.... The reduction in compressive strength and unit weight of concrete which accompanies the increase in resistance to freezing and thawing may appear disturbing at first thought for it has been generally accepted that the denser and stronger the concrete, the greater its durability [*12*]. The belief that durability increases with strength is undoubtedly true up to the point where requirements for good concrete are satisfied, but beyond this point the theory that strength is an index of durability may not be true.

water content can result from prolonged moist curing or resaturation. Therefore, the fresh unit weight should not be used as the basis of absolute volume calculations for air content of the cured concrete unless approximate values for air will suffice.

Data selected from Tables 5 and 6 of Bugg's Highway Research Board paper [*17*], while seemingly contradictory to the foregoing statement, are of interest and are given in the accompanying Table 2.

TABLE 2—UNIT WEIGHTS OF FRESH CONCRETE AND 3 BY 4 BY 16 IN. SIZE HARDENED BEAMS (AFTER BUGG [*17*]).

Aggregate Type	Condition	Air Content, %	Unit Weight of Fresh Concrete lb/ft^3	At Age 28 Days		
				Cured Unit Weight lb/ft^3	Flexural Strength, psi	Sonic $E \times 10^{-6}$
Limestone, 0.7% absorption............	vacuum saturation	3.3	148.8	148.8	825	5.52
	vaccum saturation	0.6	152.8	152.8	865	5.99
Gravel, 1.6% absorption...............	presoaked	3.6	150.1	149.7	800	5.77
	presoaked	0.3	153.9	154.1	830	6.04
	vacuum saturation	3.6	150.1	148.9	755	5.68
	vacuum saturation	0.3	154.6	153.6	870	6.11
Gravel, 1.8% absorption...............	presoaked	4.0	146.1	145.1	680	5.59
	presoaked	0.9	150.9	149.9	735	6.00
	vacuum saturation	4.8	144.4	143.8	645	5.25
	vacuum saturation	0.5	151.0	149.8	725	6.00

Vollmer [*13*] utilized fresh unit weight data to show the effect of calcium chloride admixture on the calculated air content of concretes made with Vinsol resin cement and concluded that use of calcium chloride reduced air. However, more refined tests by Pigman [*14*], Gonnerman [*15*], and others [*16*] have shown that entrained air content is increased slightly by the incorporation of calcium chloride. The density (unit weight) of *saturated* hardened concrete is consistently found to exceed that of the respective fresh concrete. This is probably attributable to two effects combined; a small reduction in the air content is usually experienced and higher

The mixtures had 3 to 4-in. slump and contained 6 sacks of Type I cement. The unit weight relationships could have been affected by variations of coarse aggregate in samples, bleeding tendency, degree of compaction, curing, and moisture condition of beams at the time of measurement. The fact that the sets of unit weight data for several mixes agree closely could be misleading, since unit weight of moist-cured specimens should be higher than that of fresh mix. In a few cases a surprising difference in unit weight is shown, corresponding to higher computed air in hardened concrete, because of variations such as loss of moisture. Axon [*18*] listed the changes in weight of hardened concretes containing varying amounts of entrained air during the progress of moist curing, water curing, and air curing; beams gained approximately 0.5 per cent in weight in 28 days of water curing and were shown to lose from 2 to 2.7 per cent in weight during 2 weeks of air drying. Since sonic E is a function of the true density of the specimen, E values in the last column of Table 2 show a reasonable relationship to hardened unit weight. It is also of interest that Bugg studied similar concretes with air contents ranging up to 7.5 per cent and concluded that no significant increases in freezing-and-thawing durability occurred with air contents in excess of 3 per cent except when very porous aggregates were placed in concrete in a highly saturated condition.

MEASUREMENT OF AIR CONTENT OF HARDENED CONCRETE

The simplest estimate of total amount of air in concrete can be derived from unit weight, provided the specimen is representative of known mixture proportions and is in a saturated state. Several papers in the 1947 ASTM Symposium on Measurement of Entrained Air [*19*] listed data for volumetric method versus the gravimetric (unit weight). Most published gravimetric tests apply to fresh concrete. Figure 3 of Swanberg's paper [*19*, p. 871], and Table X in Gregg's paper [*20*, p. 74] used unit weight test data to substantiate results by the pressure method.

In checking hardened concrete for the presence of entrained air, such as in correlation studies of pavement construction, it is not always essential to determine the size distribution of air voids when one is primarily interested in knowing the *amount* of air contained, on the assumption the void sizes are normal.

An early report by Vellines and Ason [*21*] indicated that the air voids in set concrete would respond to pressure changes, and they explained the use of an accurate timer to determine volume of total solids, V_s, in laboratory prisms 2 by 2 by 10 in. in size. By deducting V_s and volume of absorbed water from conventionally determined specimen volume, it appeared that air content could be calculated with promising accuracy.

One significant development since the publication of STP 169 in 1955 was Lindsay's report [*22*] disclosing the successful application of a high pressure meter of special design, operating at a test pressure of 5000 psi, to determine air content of hardened concrete. The equipment accommodates drilled cores up to 5 in. in diameter and 10 in. in length. Seventy-two hours oven drying with subsequent 48 hr resaturation of specimens is essential to conduct tests according to the procedure developed at Illinois Division of Highways laboratory under Larsen and Blandin. This meter is an excellent means of differentiating between concretes of different air contents, and comparisons of cylindrical specimens have shown reasonable agreement with air contents determined by microscopical means. While the elapsed time for a test is 5 days, several specimens can be processed concurrently, and it is possible to make numerous determinations in a single day. Kennedy's discussion [*22*] indicated his appreciation of this method. Subsequent tests at Waterways Experiment Station [*23*] have shown the high pressure meter to be a satisfactory tool for such tests. Quoting from Conclusion C in the report [*23*]: "Test results are qualitatively discriminating and with careful attention to . . . sources of error (etc.), results appear to be about as accurate quantitatively as are results by other methods such as microscopical examination and the low pressure testing of fresh concrete."

Supplemental Illinois Division of Highway data obtained under conditions of laboratory control are given in Table 3. Some data for 2-in.-diameter cores were omitted for brevity. However, 4-in.-diameter cores are the type usually tested in their high pressure meter. Results for the original fresh mix are compared in Table 3 to cores drilled from hardened 6 by 6 by 30-in.-beam specimens molded from the test concretes.

Erlin [*24*] has modified this apparatus to eliminate using a testing machine to apply high pressure.

TABLE 3—AIR METER TEST COMPARISONS.
Determinations on Laboratory Concrete in Plastic State and the Same Concrete in the Hardened State (after Dykins and Blandin [*25*]).
(Selected data from Ref. *25* Table 2; 4-in.-diameter cores only.)

Subgroup	Air Content in Hardened Concrete, %				Average Air Content, %	
	Individual Core Determinations				Hardened Concrete	Plastic Concrete
	GRAVEL COARSE AGGREGATE					
A........	0.5	0.3	0.1	0.1	0.2	0.2
C........	2.0	1.8	1.8	1.6	1.8	2.3
D........	3.6	3.5	3.2	2.9	3.3	3.6
E........	5.7	5.3	4.9	4.9	5.2	5.0
	CRUSHED STONE COARSE AGGREGATE					
F........	0.7	0.7	0.4	0.4	0.6	0.4
H........	3.0	2.9	2.6	2.5	2.8	2.7
I........	4.8	4.5	4.5	4.5	4.6	4.4
J........	7.1	7.0	6.8	6.7	6.9	6.5

LOSS OF ENTRAINED AIR AS SHOWN BY TESTS ON HARDENED CONCRETE

Whereas tests under closely controlled conditions may result in good agreement between measured air contents in fresh and hardened states, it is generally accepted that a loss of entrained air will be experienced between the mixing of concrete and finally placing it in the forms. Recent compilations of the State of Illinois have indicated this difference can range from a few tenths of a per cent up to nearly 1.0 per cent air.

Sweet [*26*] pointed out that the air volume in hardened material may differ from the volume of air entrained in fresh mix, due to changes in water content, concrete volume, and specific volume of ingredients. In his ASTM paper [*27*], data were presented on changes in weight (water absorbed) and unit weight in curing prior to freezing-and-thawing tests, and it was shown [*26*] that the average air content fell from an initial value of 3.0 per cent to 2.0 per cent at 28 days.

Searching for an explanation for about 20 per cent less air in hardened concrete, computed from absolute volumes, Bloem and Walker [*28*] studied the apparent specific gravity of cement in water versus time and, as a result, declared use of such specific gravity as indicated for 72 hr, the time concrete displacement data were obtained, would account for the differences found, after allowance was made for water lost in molding.

In Bureau of Reclamation tests made at about the same time, changes in theoretical unit weight of concrete with time, due to hydration, were also recognized:

Time After Mixing, hr	Theoretical Unit Weight, lb per cubic ft[a]
0..........	149.5
1..........	150.4
2..........	150.9
3..........	151.2
4..........	151.4

[a] See chart on Fig. 2 of Ref. [*29*].

When these values were applied in calculations, the agreement with the pressure test method gave evidence of accuracy.

More recently a series of American Concrete Inst. (ACI) articles by Mielenz et al [*30*] presented interesting facts on the evolution of the air void system and the possible changes which take place in freshly mixed concrete. Part I of the series covers the action of air bubbles in freshly mixed concrete, comprising a treatise on factors governing size, distribution, and stability of entrained air during hardening of the mixture. Table 4 (Part I) [*30*] includes data for concrete specimens containing two superior air entraining agents showing the air content of hardened mixture 0.5 to 1.1 per cent lower than the 5.1 per cent measured by pressure meter on the fresh mix.

TABLE 4—VOID PROPERTIES IN 3-IN. SLUMP AIR-ENTRAINED GRAVEL CONCRETES (AFTER WARREN [*36*]).

Type I Cement Mix	Darex Air-Entraining Admixture		Neutralized Vinsol Resin Admixture	
	4 sacks	7 sacks	4 sacks	7 sacks
Specific surface of voids,[a] square mm....	28.0	39.4	29.1	39.6
Air content, %......................	5.43	5.39	5.18	5.33
Average diameter of voids, mm........	0.0604	0.0669	0.0536	0.0567
Void spacing factor,[b] mm..............	0.118	0.100	0.113	0.094
Net air content, pressure meter, %.......	5.5	5.5	5.2	5.3
Fresh unit weight, lb per cubic ft........	140.3	143.1	141.1	142.8

[a] Defined as surface area of voids per cubic mm air in concrete.

[b] One half average maximum distance from void to void within paste, about 0.004 in. At a given cement content, the listed data show little difference between concretes made with the two agents. Photographs showed, at air content of 7 per cent, the air voids were more numerous and smaller in the concrete with higher cement content. Curves showed the average diameter of air voids was only slightly affected by cement content and angularity of coarse aggregate.

Bruere [*31*] (referencing the paper by Mielenz et al) studied air-entrained pastes during setting. In one of his experiments, using neutralized Vinsol resin air-entraining agent, the bubble systems of pastes of normal and accelerated setting characteristics were compared. While the case for paste-aggregate combinations was not studied, the conclusion based on Bruere's paste data was that rearrangement of bubble sizes and solution of air from small bubbles was negligible in the period during which air-entrained pastes are fluid prior to setting. By this work Bruere has provided a means whereby the size distribution of air voids in *fresh* concrete may be studied by arresting the fresh void system (introducing an agent which induces quick set) then using microscopical methods developed for samples of hardened concrete. Otherwise, it is practically impossible to determine the size distribution of air voids in tests of fresh concrete.

Powers [*32*] provides a practical insight regarding the cited papers and other related references for those who need to apply the principles of studies such as these in further investigations.

Quantitative Estimate of Air Content by ASTM Recommended Practice

Several techniques have been developed for hardened material which are based on inspection of polished sections. Verbeck [*33*] applied the camera-lucida method successfully by tracing the voids on an enlargement of a microscopic view at a magnification of about 100 diameters to determine air content at desired locations within a specimen, making comparisons with other methods. An interesting feature of Verbeck's paper was the tabulation of size and volume distribution of voids. (The nature and size range of air voids in Types I and IA cement concretes were shown clearly in camera-lucida drawings in Klieger's [*34*] paper.) The published discussion of Verbeck's paper and subsequent developments have shown that skilled technicians at the laboratories of the Corps of Engineers, the Missouri Highway Department, and elsewhere were meeting the need for specialized microscopical examination of hardened air-entrained concrete; this is discussed by other authors in this publication.[6] With the additional evidence provided by new techniques [*35*] for determining the nature of air voids, Powers was able to advance the theory of permissible spacing to show that, for high resistance to freezing and thawing in cement pastes, the average void-to-void distance should be about 0.02 in. or less.

The results reported by Warren [*36*] at the University of Wisconsin include photomicrographs of carefully polished interior sections of air-entrained concrete magnified to 40 diameters. Voids filled with fluorescent dye in Canada balsam were photographed in ultraviolet light. Void properties, calculated from measurements by the plane intercept method, are interesting because those introduced by two accepted air-entraining agents had essentially the same specific surface, as is seen in Table 4.

ASTM Recommended Practice for Microscopical Determination of Air Void Content, Specific Surface, and Spacing Factor of the Air Void System in Hardened Concrete (C 457 – 60 T) covers procedures for both linear traverse and point-count methods. The procedures are not simple, requiring skill and experience to achieve desired accuracy. The recommended practice is replete with specific instructions for proper conduct of the microscopical examination and includes numerous notes explaining special details. Therefore, it is rather extensive. Stated briefly, the technique is based on preparation of representative plane interior surfaces by sawing and fine grinding on suitable laps, then traversing the prepared surface under a microscope to determine the relative amount, size, and spacing of air voids present. The ability to distinguish air voids is dependent of the qualities of the prepared surface, locating a typical field for scanning, effect of magnification, and the operator's skill using stereoscopic binocular with the surface illuminated from one direction at a low angle. In this way the air voids are shadowed and recognizable down to 15 μ size or less;[7] low incident light (around 30 deg from horizontal) also aids in detecting air voids, shallow because the cut surface may be close to the bottom of the void. In determinations of void systems of high specific surface better perception at higher magnification will substantially increase the air determined microscopically. Although this procedure requires skill and precision and capable technicians, it is conducted regularly in research studies and has proven to be a practical method for study of job concrete. In fact, such procedures are the only methods available for the determination of α, the specific surface of air voids, and $\bar{L}$, the computed spacing factor.

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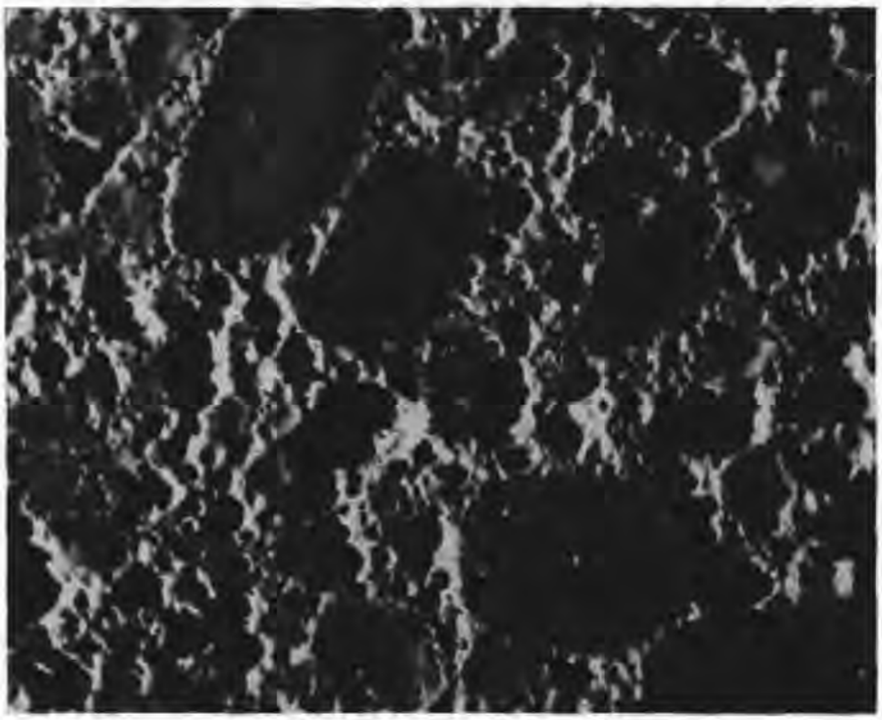
[6] See p. 125 and p. 381.

[7] For details see ASTM Recommended Practice C 457 – 60 T, Section 5(b).

CONTRAST OF PURPOSE FOR HIGHER LEVELS OF AIR INCORPORATED IN CONCRETE

Usually the amount of entrained air in hardened concrete is intended to be within the range of 3 to 7 per cent in the form of minute air voids of high specific surface (ranging 700 to 1400 in.^{2}/in.^{3}), in order to minimize the sacrifice of mechanical properties and insure satisfactory durability. On the other hand the inclusion of air in excess of 8 per cent will markedly reduce compressive strength. Figure 2 is a photomicrograph of a concrete containing air voids at the high level of 30 per cent which would be prohibitive from the structural standpoint but commonplace if cellular concrete were desired. This figure forms an interesting contrast with the illustrations on p. 262 of August, 1958 issue of *Journal*, American Concrete Inst. [*30*] presented by Mielenz et al to show normal void systems of concrete with 3.5 per cent air.



Courtesy Master Builders Div., Martin-Marietta Corp.

FIG. 2—Concrete containing excessive entrained air (×20). Air determined microscopically to be approximately 30 per cent.

Polished sections of concrete with high air content, or weak for other reasons, present problems in surface preparation. According to Mielenz and Wolkodoff: ". . . at very high air content the bubbles tend to coalesce, and the thin septa and ridges between the voids are easily lost during grinding," adding the thought these factors would impair the accuracy of the linear traverse technique.

Thus there are two main purposes for incorporating air in concrete:

1. Air entrainment for frost resistance, reduced bleeding, and improved workability. If properly controlled, the voids will be of high specific surface and provide the required spacing factor of 0.008 in. or less.

2. Large amounts of air, introduced

by aeration or generation of gas, in the range 10 to 30 per cent or more air content, primarily to reduce the density of the concrete product. For purposes of insulation and weight reduction the void size is not as critical as in structural applications.

Referring to the "non-AE" subclass mentioned earlier, in the explanation of Fig. 1, the smaller amount of air present in nonair-entrained concretes is known as incidental air, or as entrapped air, or accidental air. In normal weight concrete this "incidental" entrained air is less than 2 per cent, although higher values may result from inadequate compaction. The amount of incidentally entrained air is dependent on mix proportions, aggregate particle shape, aggregate absorption, amount of fines, organic impurities, and other minor factors.

DETERMINATION OF UNIT WEIGHT USING STANDARDIZED PROCEDURES

Calibrated standard cylindrical measures are used under Method C 138 to determine the unit weight of freshly mixed concrete. Theoretical weight per cubic foot, computed on an air-free basis, was mentioned previously. When batch proportions and weights of ingredients are accurately known the air content is determinable by the method of absolute volumes—deducting the volume of component ingredients from the calculated volume of the batch.

Where composition is known, some predictions of the air dry unit weight can be made or calculated by making certain assumptions. Such predictions are dependent on the accumulation of data pertaining to equilibrium air dry weight at a given relative humidity. To a certain extent air dry unit weight values, while fictitious in nature, are useful in dead-load computations.

Recently ASTM accepted Tentative Method C 567 – 65 T, Unit Weight of Structural Lightweight Concrete, which includes the testing of hardened concrete by determining the air dry weight of standard 6 by 12 in. cylinders moist cured 7 days and air dried at 50 relative humidity for 21 days. As mentioned in the introduction of this paper, specimen volume is determined in Method C 567 by making suspended-immersed weighings. Notably the procedure given in Section 4 of this method is applicable to normal weight concrete specimens, too. Similar displacement procedures are available for tests of concrete masonry units under ASTM Method of Sampling and Testing Concrete Masonry Units (C 140); however, Method C 140 specifies a draining period of 1 min which is required to properly calculate the effective specimen volume. The short period required for completing the "saturated surface dry" and "immersed" weighings minimizes the possibility of error in determined volume due to further reabsorption, unless the specimen is not immersed for the required 24 hr period. On the other hand, the 1-min drain time tends to increase the calculated volume of a porous concrete, as in the case of a lightweight concrete masonry unit, where increased drainage time of 5 or 10 min could have the effect of reducing the apparent volume of the specimen by 1 or 2 per cent.

The same type displacement procedures may be used to determine volume and unit weight of specimens of various sizes obtained from hardened concrete according to ASTM Methods of Obtaining and Testing Drilled Cores and Sawed Beams of Concrete (C 42). Having obtained physical data on a given concrete sample, an analytical approach can be made according to the method of absolute volumes by establishing the proportions and specific gravity of ingredients. This principle has been used in our laboratory to indicate the absence of entrained air. By measuring the volume and weight of a suitable resaturated specimen, the concrete may be disassembled after dehydration at 600 C in the manner described by Blackman [*37*], and since the proportions of dry solids are determinable, unit air and water contents may be calculated with reasonable accuracy. Axon [*38*] developed and published a procedure for determination of air content and estimation of original unit water content based on physical and microscopical methods.

TABLE 5—ABSORPTION OF AIR-DRY CONCRETE (AFTER KLIEGER [*40*]).

Cement	Coarse Aggregate	Slump, in.	Absorption, %, by 72-hr Resaturation of Air-Dry Concrete								
			4-Sack Mixes			5½-Sack Mixes			7-Sack Mixes		
			¾ in., max	1½ in., max	2½ in., max	¾ in., max	1½ in., max	2½ in., max	¾ in., max	1½ in., max	2½ in., max
Type I.....	gravel	3	3.4	2.7	2.7	1.6	1.5	1.6	1.2	1.1	1.2
Type IA....	gravel	3	3.1	2.5	2.5	1.7	1.4	1.4	1.2	1.1	1.1
Type I.....	stone	6	4.4	3.3	...	2.6	2.2	...	1.7	1.6	...
Type IA....	stone	6	3.6	2.9	...	2.2	2.0	...	1.7	1.6	...

TABLE 6—TESTS OF 3 BY 6-IN. VIBRATAMP CONCRETE CYLINDERS.

Laboratory Mix	Quantities, lb per cubic ft (compacted)							Yield, Blocks, per sack[a]	Compressive Strength Air Dry at 28 Days, psi (7 days moist)
	Type III Cement	Water	Dry Admixture	Fine Natural Sand	Fine Aggregate Through No. 8 Sieve	Coarse Aggregate Retained on No. 8 Sieve	Total Concrete Fresh Density		
A.....	12.5	13.7	0.008	6.6	23.3	33.2	89.3	25.1	1350
B[b].....	12.6	13.8	0.008	6.7	23.5	33.6	90.2	24.9	1620[b]
C.....	12.6	13.8	0.016	6.7	23.6	33.7	90.4	24.8	1470

[a] Calculated on basis of nominal block volume of 0.30 ft^3.

[b] Relatively larger machine-mixed batch, other batches hand-mixed.

ENTRAINED AIR AND RELATED PROPERTIES

Though affected also by coarse aggregate character, the absorption of concrete is primarily dependent on the quantity and quality of paste or mortar component and for given materials absorption is a useful criterion of quality. Hansen [*39*] published data showing the effects of overrodding on 2½-in.-max crushed stone concrete made with different sands and cements, including air-entraining cement. Table V of Ref. [*2*] included interesting saturated surface dry unit weight and absorption data of resaturated air dry concrete, as abridged from Hansen's paper.

In discussing reabsorption of water, the more recent data of Klieger [*40*], shown in Table 5, representing a wider variety of proportions, are of interest because the sand was reduced to compensate for the increased amounts of air. The selected results show reduction of absorption in lean mixes where there was a marked reduction of mixing water when air was entrained.

Following the line of paradoxes expressed in a paper by Gilkey [*41*] which, incidentally, traced the relationships of voids, density, and air content—it is appropriate to emphasize that air-entraining cement will increase consolidation of concrete block mixtures of dry tamp consistency. Swayze [*42*] mentioned this in an article in *Civil Engineering* when "treated" cement was being introduced to the block industry, and Helms and Bowman [*43*] presented quantitative data. Results supporting this principle are given in Table 6 for laboratory specimens containing expanded slag aggregate, including a double dosage of dry powdered air-entraining agent which, in plastic concrete, could cause an inordinately high air content.

VARIED AMOUNTS OF VOIDS AND SPECIAL PURPOSE CONCRETES

Methods used in commercial practice for imparting dense structure to concrete by manipulative means include: (1) production of hard floor finishes from dry-tamped concrete using power floats [*44*], (2) densification of initially overwet concrete by vacuum techniques to achieve high early strength, (3) making of concrete pipe by spinning and packer methods, and (4) manufacture of lightweight masonry units [*45*].

Numerous reports have traced the development of the use of gas-forming agents, such as aluminum powder and hydrogen peroxide, to make lightweight compositions. Aluminum powder has been used for many years in other countries such as Sweden, and more recently in Canada, to manufacture cellular products under familiar trade names (Siporex, Durox, and Ytong) but these products have not proved feasible for production in our country. Manufacturing problems and economic factors combined may be the reasons which have delayed its acceptance here. Erratic behavior of aluminum powder dependent on the sensitivity to chemical properties of cement and difficulty in distributing the powder to get uniform expansion are among the factors to be considered. Foamed concretes that use large amounts of air-entraining agent as well as gas concretes are likely to show high shrinkage, and stabilization by autoclaving is desirable. Lightweight cellular concrete [*46*] is discussed more fully in the paper by Lewis[8] in this publication.

In the range of lowest density concretes, usually made with lightweight aggregates, an intended use for thermal insulation often makes strength a secondary consideration. The criterion of strength-weight ratio may be used, however, to rate the efficiency of compositions of similar unit weight. The principle of "no fines" concrete [*47*] could even be modified to the use of lightweight aggregate and foamed paste to produce a variety of densities down to the low limit. Low specific weight is dependent on the presence of voids, whether formed by air or water, as holes in honeycomb or as pores in the aggregate. The insulating efficiency of concrete may be correlated closely with the unit weight of the oven-dry specimens and the compressive strength with air-dry density. Although slight advantages can be claimed for certain aggregates in weaker, leaner mixtures, the plotted thermal conductivity in Kluge's [*48*] Fig. 14 permits the citation of *K* values which could be assumed applicable to any oven-dry lightweight concrete mix with low unit weight:

Oven-Dry[a] Unit Weight of Concrete, lb per cubic ft	Conservative Maximum Thermal *K* Value[b]
20..........	0.7
42..........	1.5
55..........	2.0
70..........	2.5

[a] Slight advantages for certain combinations could be overshadowed by the effect of small amounts of moisture retained by air-dry specimens.

[b] Less conservative values may be found in Neher's Fig. 3, *Journal*, Am. Concrete Inst., September, 1951, p. 68.

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[8] See p. 359.

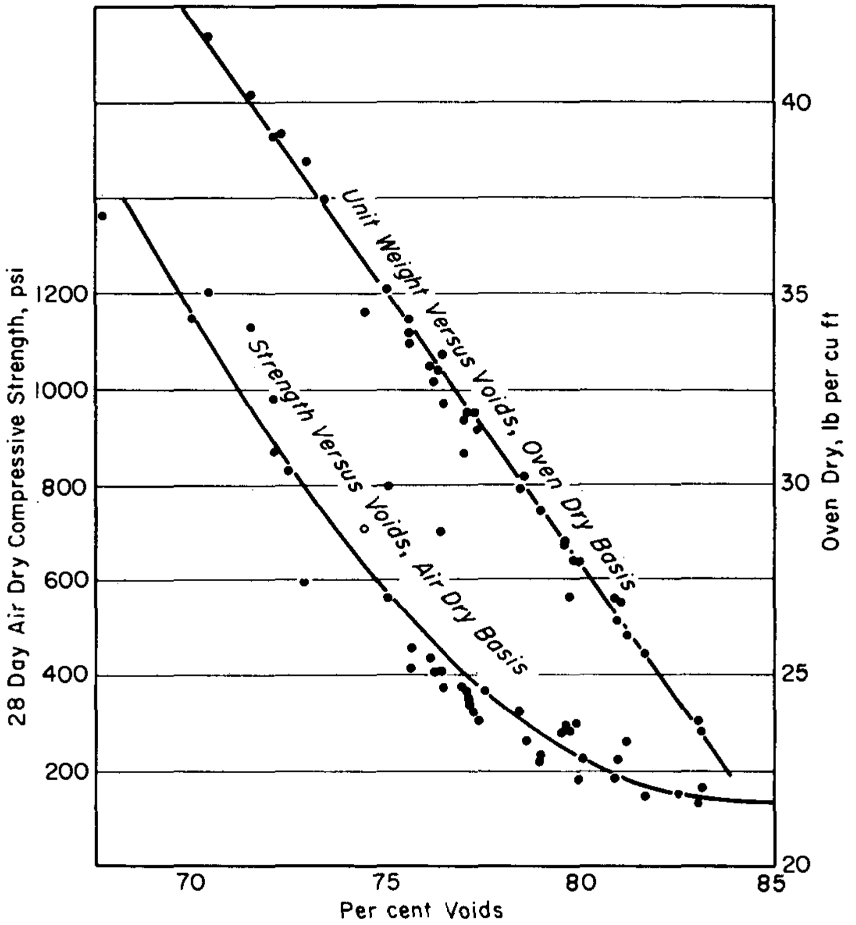

FIG. 3—Relationship of unit weight and strength to per cent voids, Type III cement insulating concrete made with expanded perlite aggregate.

TABLE 7—ABRIDGED AVERAGE DATA FROM KEYSER'S TABLE I [*49*] 6-SACK 1½-IN.-MAX IRON ORE CONCRETES, PROBABLY NO-SLUMP.

	Quantities per cubic yard, lb			
	11[a]	12[a]	13[a]	14[a]
Cement	563	563	563	563
Water	317	280	317	224
Sand	383	...	...	744
Fine iron ore	876	789	1627	...
Coarse iron ore	2687	2670	3204	3487
Rivet punchings[b]	2553	3069	1667	2342
Total	7379	7371	7378	7360
Design unit weight, lb/ft^3	273.3	273.0	273.3	272.6
Compressive strength, psi, 28 days	2230	2190	2750	4260
Unit weight, lb/ft^3, 28 days	270.5	272.1	268.6	271.8

[a] Reference mix number.

[b] Sized ½ to 1 in. in diameter and ¼ to ⅝ in. thick; oil and rust free.

The plotted data of expanded perlite concretes in Fig. 3, obtained in 1948, illustrate the relations of voids to weight and total solid volume to compressive strength. Results for 40 test combinations are shown. These mixes include three aggregate samples, three volume proportions of aggregate, and usually either an air-entraining agent or a fine plasticizing admixture. The curve of strength versus solids has limitations, since cement content is reflected only as weight, but it has unique applicability in the extreme range of high voids.

At the other extreme, the utilization of heavy aggregate concrete because of its weight is of direct engineering importance. Much ordinary concrete, used in gravity dams, lock walls, counterweights, and some foundations, is dependent on the weight of the mass for its utility.

Keyser studied heavy aggregate concrete for making counter weights for bascule bridge as far back as 1932. Although not directed toward nuclear application, the mixtures described contained iron ore and steel punchings which have been used as aggregates for radiation shielding. In order to avoid segregation the quantity of mortar used was only slightly greater than that required to fill the voids in the coarse aggregates. Revised compositional data selected from the final mixtures, listed in Table 7, are of historical interest.

The special concretes used for radiation shielding require dense aggregate materials such as iron ores; for this purpose Callan [*50*] described high unit weight concretes in the range of 225 to 275 lb/ft^3. This topic is covered adequately in Polivka's[9] paper. Concrete in the weight range above 250 lb/ft^3 has been used extensively for permanent ballast in ships [*51*].

In conclusion, it should be mentioned that unit weight tests are the time-honored basis for determination of yield,[4] and sometimes it is expedient, as for the data plotted in Fig. 3, to calculate this information from weight and displacement or weight and dimensions of hardened specimens. The determination of yield and interdependent unit contents of cement and water is necessary in both laboratory and field for the proper control of concrete mixtures.

When air is incorporated for the purpose of reducing density, the size of voids is of little consequence if they are uniformly distributed throughout the mix. But the size of air voids entrained to improve durability is extremely important, and it has been realized for many years that if air is efficiently dispersed into the finest attainable size a smaller amount will be required for durability with subsequently less effect on strength.

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Hardened Concrete

CEMENT CONTENT

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Determination of the portland cement content of hardened concrete is often a matter of concern to engineers and chemists working with mortars and concretes. Unfortunately, a precise method of analysis has not been developed due to the many complexities of the material under test. Difficulties include the selection of representative samples, the presence of aggregate materials from unknown sources, the use of portland cements that differ substantially from an average composition, the presence of admixtures or additions (such as pozzolans or finely ground slag), the degree of hydration of the cement, the effect of carbonation as contrasted with hydration, and the effect of leaching of the concrete.

In view of such formidable obstacles, chemical procedures demand that every effort should be made to obtain samples of the materials used in the concrete mixture. Where adequate information is obtained on the coarase and fine aggregate and cementing material, a reasonably accurate estimate of the cement content of hardened concrete can be made by chemical methods. In the absence of sufficient information on the source materials, assumptions must be made with respect to the amount of soluble substances that may be contributed to the analysis by the aggregate fractions and with respect to the ratio of aggregate materials present in the mixture. The assumptions affect the reliability of the test results. In some instances, the use of several schemes of analysis should be considered since the information derived from one method may assist in interpreting the results derived from an alternate method.

This report outlines several methods that have been employed to determine the cement content of hardened concrete and indicates the reliability of the results that have been obtained, with emphasis being given to the procedure that is available as ASTM Test for Cement Content of Hardened Portland Cement Concrete (C 85). Results of cooperative tests undertaken by ASTM Committee C-9 to evaluate the reliability of the ASTM method of test are presented.

Primary Factors Relating to the Composition of Concrete

Published investigations on methods to determine cement content of hardened concrete emphasize the importance of obtaining background information on the ingredients within the test sample. A brief summary of the main items involved in establishing a basis for a test procedure are presented below.

Portland Cement:

Where the type of cement is not known, it is generally recommended that the composition of a typical Type I portland cement serve as the basis for

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chemical calculations. Most investigators have used a soluble silica content of 21.75 per cent and a soluble calcium oxide content of 63.0 per cent [*1*].[2] These are reasonably good values to employ in the determination, but many modern portland cements are known to contain more calcium oxide and less silica. These values refer to cement in unhydrated form.

Two general approaches to the calculation of cement content are used; the first is based on the original mix proportions at the time the concrete was produced and the second relates to the cement content of the concrete after aging. In the first instance no effort is made to incorporate the water of hydration in the calculation. In the second instance the water of hydration becomes part of the cement content. Calculation based on a cement "factor," or on the amount of unhydrated cement per cubic yard of plastic concrete, is generally recommended.

Water:

The water content of concrete is of considerably importance in determining the cement content, and most of the test methods are directly involved with this consideration. Unfortunately, the determination of the water content of a cement paste in hardened concrete is a complex problem, since the process of aging of concrete involves reactions between the anhydrous cement and the mixing water both of a chemical nature (such as production of hydration products) and of a physical nature (such as absorbed and adsorbed water). Furthermore, in time, some of the mixing water is lost with the formation of fine voids which may or may not be capable of resaturation. These voids differ from those which are present as the result of air or gas entrainment.

Water is present in hardened concrete in several forms. Part of the original mixing water is lost through bleeding of the plastic concrete and by evaporation; some water may also be lost as a result of carbonation of hydration products during the aging period. In addition, water contents may increase with aging due to hydration of cement [*2*].

While most investigators have taken into account the need for considering the water of hydration in carrying out the analysis of concrete, there is as yet no agreement as to how this should be done. It has been suggested, for example, that the sample of concrete be initially heated to an elevated temperature prior to making the cement determination to remove all combined water. Other methods employ a fixed estimate of the water content in the hardened paste. Powers has suggested a maximum water of hydration in hardened neat cement paste of 26 per cent by weight of anhydrous cement [*2*]. More recently Yaltkaya has suggested an average value of 14.1 per cent [*3*]. A few investigators have attempted to estimate the original water content of the concrete mixture by establishing the density of test specimens when in saturated condition [*4–6*]; this density value may also be used to relate the analytically determined cement content to sacks of cement per cubic yard in the original mixture.

While all of these procedures are of value to the analyst, none is sufficiently precise to give an accurate measure of the original water content of the mixture.

Voids:

In making analyses of hardened concrete by the use of microscopical procedures, a determination of air-void content may be useful in evaluating the

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

TABLE 1—SOLUBILITY OF COMMON ROCK-FORMING MINERALS.

A. Minerals Relatively Insoluble in HCl

- Feldspars:
  - Orthoclase (microcline)
  - Anorthoclase
  - Plagioclase:
    - Albite
    - Oligoclase
- Muscovite
- Pyroxene var. augite
- Amphibole, hornblende
- Garnet
- Quartz
- Flint
- Pyrite
- Talc
- Enstatite (bronzite)

B. Minerals Partly or Wholly Soluble in HCl

- Feldspars:
  - Plagioclase:
    - Andesine
    - Labradorite
- Epidote
- Hematite
- Magnetite
- Limonite
- Pyrrhotite
- Calcite
- Dolomite
- Gypsum
- Apatite

C. Minerals Which Gelatinize in HCl

- Feldspars:
  - Plagioclase:
    - Bytownite
    - Anorthite
- Nephelite
- Olivine (chrysolite)
- Analcite
- Sodalite

D. Minerals Insoluble in HCl But Which May Be Decomposed in Part (are decomposed by H_2SO_4)

- Biotite
- Phlogopite
- Serpentine

TABLE 2—SOLUBILITY OF COMMON ROCKS.

A. Rocks Relatively Inert to HCl

- Granite
- Pyroxenite
- Pegmatite
- Obsidian
- Pumice
- Perlite
- Felsites:
  - Rhyolite
  - Trachyte
- Silica bonded conglomerate
- Silica bonded sandstone
- Silica bonded quartzite
- Shists
- Red Slate
- Gneiss
- Chert
- Arenaceous shale
- Carbonaceous shale
- Phyllites
- Amphibolite

B. Rocks Partly Soluble in HCl—Releasing SiO_2

- Syenite
- Diorite
- Gabbro
- Peridotite
- Diabase } "trap rock"
- Basalt }
- Felsites:
  - Dacite
  - Andesite
- Ferruginous conglomerate
- Ferruginous sandstone
- Ferruginous quartzite
- Arkose
- Graywacke
- Gypsum
- Serpentine

C. Rocks Partly or Wholly Soluble in HCl—Releasing CaO

- Limestone
- Dolomite
- Marble
- Limestone conglomerate
- Sandstones bonded with calcite or dolomite
- Quartzite bonded with calcite or dolomite
- Graywacke—calcite bonded
- Gray slate
- Calcite bonded shales

cement content of the concrete (Recommended Practice for Microscopical Determination of Air-Void Content, Specific Surface, and Spacing Factor of the Air-Void System in Hardened Concrete (C 457 – 60 T).) Unfortunately, the air content of the hardened concrete may be significantly different from that which was present at the time the concrete was placed. It may, for example, be substantially lower than that indicated by standard tests on the fresh concrete if the amount of compaction which was used in the placing of the concrete was greater than that used in the original air-content determination. Conversely, the air content of hardened concrete may be higher, reflecting variations in water content and fluidity of the concrete. The voids observed in hardened concrete differ in their size, frequency of occurrence, and distribution. They include voids resulting from purposeful "entrainment" and those resulting from mechanical "entrapment" at the time the concrete was placed.

Air content of hardened concrete varies from place to place in the concrete depending upon the circumstances prevailing during the placing and finishing of the concrete. Thus, pronounced bleeding or settlement of the plastic concrete may result in irregular distribution of air, in the formation of relatively large voids resulting from channeling of water, and entrapment of water under coarse aggregate particles.

Aggregate:

Where a chemical method is employed in analyzing hardened concrete, the type of aggregate should be identified. In fact, the choice of the method of analysis may depend upon this identification.

Whether or not the aggregate will be attacked by hydrochloric acid is dependent on the action of the acid on the minerals and amorphous phases composing the rock. Table 1 lists common rock-forming minerals according to their reaction with hydrochloric acid (HCl). Based on these data, rocks in common use are grouped according to their reaction to hydrochloric acid in Table 2. A simplified tabulation of rocks such as given in Table 2 cannot be entirely complete and unambiguous. For example, there are some alkaline and basic volcanic rocks which contain zeolites, and there are cherts and shales which include significant proportions of calcite or dolomite in their compositions. Such rocks may, therefore, be subject to some attack by HCl. Artificial aggregates, such as slags and lightweight aggregates, are usually easy to identify and must be tested separately for their solubility in hydrochloric acid as these materials vary significantly in their composition. It is recommended that wherever possible the aggregate be subjected to petrographic inspection in order to give the analyst making the chemical investigation a better understanding of possible extraneous sources of soluble silica or calcium oxide.

Since many aggregates can be expected to introduce soluble silica or calcium oxide into the analysis, suitable corrections must be made in the calculation for cement content to compensate for this contamination. In general, the method of calculation should be established on the basis which involves the less soluble constituent in the aggregate. For instance, if the aggregate is a limestone, the use of calculations based on the silica content of the cement is to be preferred since only a very small correction will be made as a result of the soluble silica fraction of the limestone aggregate. ASTM Method C 85 – 54 requires that wherever the original aggregates are available, blank tests shall be run to determine the exact amounts of silica and calcium oxide which are soluble under the condition of the test.

In making the correction for aggregate solubility, it is desirable to ascertain the proportion of fine and coarse aggregate used in the concrete mixture. Where this is not known it may be determined by microscopical techniques, or a concrete proportion of 1 part cement, 2 parts fine aggregate, and 4 parts coarse aggregate by weight may be assumed. These proportions represent a typical concrete mix design and, where the aggregate solubilities are small, the corrections made in the calculations for cement content are reasonably accurate. ASTM Subcommittee III-1 of Committee C-9 is working on revisions of Method C 85 – 54 which are intended to make the determination of aggregate proportions unnecessary. (This procedure is discussed later.)

SAMPLE SELECTION AND PREPARATION

The selection of representative samples of hardened concrete for test purposes is no simple matter. Variations in concrete from a given source can usually be expected depending on the precise location of the test sample, the depth at which taken, the conditions of exposure, the possibility of the presence of contaminating materials, and so on. Samples of concrete taken from a distressed area may prove to be quite different from those taken from other locations, and such samples should be kept apart with proper identifying marks to assist the laboratory investigator. In general, a well planned program of sample selection should be considered and, if possible, a statistical evaluation should be used. Preferably the specimens should be 6-in.-diameter cores and should be forwarded to the laboratory intact to assure that suitable physical examinations can be made.

Preparation of Sample for Physical Analysis:

Three approaches for determining the basic constituents of concrete by physical techniques are presented in the literature. They consist of microscopical analysis, physical determination of chemical compounds or composition by means of neutron activation, and neutron scattering techniques.

In the case of microscopical analysis, for example as employed in ASTM Recommended Practice C 457, the preferred procedure is to prepare a cut, flat lapped surface which may be obtained by sawing a concrete core lengthwise and carefully grinding and polishing the exposed surface.

In the work carried out by Covault and Poovey [7], samples were prepared for neutron activation analysis by screening fresh concrete through a No. 4 sieve and preparing small samples of mortars from the screened concrete. Most of the work conducted in this study dealt with uniformity of mixing fresh concrete, but similar techniques might be applied to hardened concrete.

Neutron scattering techniques employed by Yaltkaya et al [3] involved estimation of the water content of oven-dried samples of powdered concrete. The authors suggest that the method is applicable to nondestructive techniques when corrections are made for capillary water *in situ.*

Preparation of Sample for Chemical Analysis:

Proper preparation of the sample for chemical analysis is one of the most important factors determining the reliability of the test method. A sample of no less than 10 lb of the hardened concrete should be employed for each test, and the entire sample should be used in the preparation of the fractions used in the analysis.

The sample may be broken up and crushed in a suitable machine to about ½ in. in size and further reduced to a fineness of approximately 150 to 200 mesh in a ballmill, disk pulverizer, or other device (ASTM Method C 85 – 54). Modifications of Method C 85 – 54 being studied in ASTM involve an initial heating of the sample to a temperature of 500 to 600 C. Heating destroys the strength of the concrete and makes it possible physically to separate coarse aggregate from the sand and cementitious material. In this instance the cement content is determined on the mortar fraction. In either case, after preparation of the fine material for test, the sample should be thoroughly mixed and quartered and a portion approximating 100 g taken for test. The test sample should be carefully freed by means of a strong magnet from particles of metallic iron introduced from the pulverizer or ballmill used in the grinding operation. After grinding, the sample should be dried at 105 C for at least 2 hr prior to test.

Interlaboratory studies indicate that grinding the samples may result in a significant dust loss. Since the dust is rich in cement, erroneous (usually lower) results have been obtained.

The method of sample preparation which involves heating of the sample and subsequent separation of aggregate is potentially a preferred procedure in that it circumvents the necessity for making assumptions related to the proportions of the aggregate materials present in the concrete, restricts the problem of aggregate solubility to the fine aggregate fraction by physical separation, and simplifies the calculations by removing the water of hydration from the mix.

METHODS OF ANALYSIS, SELECTED RESULTS, AND OBSERVATIONS

The various methods for determining portland cement content in hardened concrete employ procedures which, in several instances, were not specifically designed for such use. For instance, microscopical measurements are generally concerned with determining the volumetric proportion of aggregate, cement paste, and air voids. However, several investigators have attempted to extend the microscopical procedures in and effort to relate them specifically to the determination of portland cement *per se*. The purpose of this section is to describe the techniques of analysis that have been used for this purpose with specific emphasis being placed on the modifications or interpretations that have been considered by the investigators.

Microscopical Techniques:

During the last several years increasing interest has been shown in methods based upon physical examination of concrete surfaces. Basically, these methods have been adopted from procedures used in petrography. The methods depend on the fact that if a plane or a line is passed through an aggregation of heterogeneous material oriented at random, the total linear intercepts of each constituent with that plane or line are proportional to the volumes of the respective constituents. There are two general procedures as outlined below:

(*a*) The linear traverse method employs a continuous scanning of the surface of the material and while so doing integrates the lengths of the separate classes of particles, such as, in the case of concrete, large aggregate, small aggregate, and air voids. The volume of the constituents is determined mathematically, and the cement paste volume is calculated by subtracting this total volume from the whole.

(*b*) The point count method biaxially scans the specimen in a series of steps of equal intervals. The number of times that the intercepts of this chosen network intersect a constituent is recorded and from the frequency with which areas of each component coincide with this network, the volume of the component in the specimen can be computed.

These methods have been used for the determination of air voids in hardened concrete under ASTM Designation C 457 – 60 T. The methods have also been extended to include fine aggregate, coarse aggregate, and cement paste by Polivka et al [*8*] and by Axon [*9*]. The results of the latter investigations indicate on the average that a good correlation may be obtained between the measured paste content and the actual cement content determined from a calculation involving the original water and portland cement contents after adjustment for losses due to bleeding. However, the paste content is not necessarily a direct measure of the cement content of the concrete, since the difference in density of water as compared with portland cement results in a substantial change in the paste content with a relatively small change in water content. These procedures, therefore, may have limited value as a means of specifically measuring the portland cement content for all samples of concrete.

TABLE 3—RELATIVE ERROR IN PORTLAND CEMENT DETERMINATION ON HARDENED CONCRETE (POLIVKA et al)

NOTE—The relative error is determined from the difference between the determined and known cement content divided by the known cement content.

Known Cement Content	Microscope Method	ASTM Method C 85 – 54, Relative Error, %			
		Solution SiO_2 Method		Solution CaO Method	
	Relative Error, %	Materials Not Available	Materials Available	Materials Not Available	Materials Available
7.5	+10.7	+77.5	+54.7	−6.7	+13.3
9.8	+4.0	+63.3	+41.8	+6.1	+9.2
11.5	+5.7	+20.9	+4.3	−11.3	+1.7
12.5	+8.5	−17.6	−28.8	−9.6	+1.6
12.5	+13.0	+6.5	−8.0	−13.6	−1.6
13.7	−2.8	+25.5	+8.8	+1.5	+8.8
Maximum error, %	13.0	77.5	54.7	13.6	13.3
Average error, %	7.4	35.2	24.4	8.1	6.0

Both the linear traverse method and the point count method have a long history of successful use in petrography. Selected references for background of linear traverse and point count analyses are given in references [*10–13*]. The investigation reported by Chayes and Fairbairn [*13*] gives a comprehensive analysis of the reliability of the point-count method. A disadvantage of these methods as related to concrete is that a specialized traveling microscope or travelling stage device is required. The apparatus described in ASTM Recommended Practice C 457 may be adapted for this work by simple modification to accommodate the recording of traverse lengths.

Microscopical analyses made by Polivka et al [*8*] are shown in Table 3 in which comparisons are made by the investigators of the relative error of the microscopical method with the errors of chemical methods. In making these determinations Polivka et al assumed an average value of the ratio of hydration water to cement of 20 per cent which he derived from the work of Powers and Brownyard [*14*]. The specific gravities of portland cement and of nonevaporable water were assumed to be 3.15 and 1.0, respectively. The porosity of the paste and the volume of submicroscopic voids were ignored. While these assumptions have been subject to question [*15*], it is evident that the microscopical method as conducted by these investigators compared favorably with the best results obtained using the chemical methods. All aggregates used were predominantly siliceous; hence, the results of the chemical test should be reported on the soluble calcium oxide basis.

Neutron Scattering Techniques:

Recently several investigators have employed the techniques involving neutron stimulation of hardened concrete samples for estimating the cement content. Yaltkaya et al [*3*] employ a method of nondestructive testing which is based on a determination of over-all water content (capillary water plus water of hydration). These investigators estimate the capillary water by the use of electrical conductance and the total water by neutron scattering technique. The water of hydration is then determined by subtraction.

Based on the investigation of 64 samples of cement, a value of 14.1 per cent is used by these authors as the mean percentage of hydration water in cement. The cement content of hardened concrete may be calculated from this value and from the amount of hydration water which is determined to be in the concrete in question. The method has not been investigated in the field as yet but does show interesting possibilities.

TABLE 4—PROPORTIONING OF CONCRETE USED BY FORD [*17*] IN EVALUATION OF CHEMICAL METHODS FOR ESTIMATING CEMENT CONTENT OF HARDENED CONCRETE.

Ref. No.	Mix by Weight	Known Cement Content	Aggregates
1........	1:1:2	32.1	siliceous with some limestone (Elgin sand and gravel)
2........	1:3.2:4.8	15.0	
3........	1:1:2	24.8	siliceous (Cow Bay sand and gravel)
4........	1:3.2:4.8	11.1	
5........	1:1:2	38.4	limestone (screenings and crushed)
6........	1:4:4	19.1	
7........	1:3	24.8	siliceous (graded Ottawa sand)
8........	1:8	10.7	

Covault and Poovey [*7*] used neutron activation of portland cement concrete in an effort to establish the calcium content resulting from the portland cement. Cast samples of portland cement mortar were activated by means of a one million volt Van de Graaff positive ion accelerator. The amount of radioactivity produced in the sample of mortar was found to be directly proportional to its portland cement content. The method is based on the ability of the neutron to produce radioactive calcium under the conditions of the experiments and is limited to specimens which contain aggregates that are either essentially free of calcium content or are of known calcium content. Should this method develop as a valid procedure, it is conceivable that normal X-ray spectrographic techniques may also be of interest.

Chemical Methods:

Nearly all of the chemical procedures are based on the determination of soluble silica or soluble calcium oxide or both under controlled conditions of leaching either with acid solutions or, in the case of silica, with acid and alkali. The selection of the particular method depends largely on the aggregate con-

TABLE 5—DETERMINED CEMENT CONTENTS OF EIGHT HARDENED CONCRETES AND MORTARS (FORD).

Based on Per Cent of Known Cement Content

NOTE—Method I is based on ASTM Method C 85 – 42 except that both acid and alkali soluble silica are determined.
Method II is similar to Method I except that different techniques are used to digest and filter samples.
Method III is based on silica and calcium oxide determinations and also employs minor changes in techniques.

Ref. No.	Method I						Method II						Method III					
	Uncorrected for Aggregate			Corrected for Aggregate			Uncorrected for Aggregate			Corrected for Aggregate			Uncorrected for Aggregate			Corrected for Aggregate		
	Acid Solution SiO_2	Alk. Solution SiO_2	Total Solution SiO_2	Acid Solution SiO_2	Alk. Solution SiO_2	Total Solution SiO_2	Acid Solution SiO_2	Alk. Solution SiO_2	Total Solution SiO_2	Acid Solution SiO_2	Alk. Solution SiO_2	Total Solution SiO_2	Total Solution SiO_2	Total Solution CaO	Avg	Total Solution SiO_2	Total Solution CaO	Avg
1	90	15	105	86	6	92	95	12	108	90	4	95	95	...	...	90	...	...
2	90	28	118	79	3	82	90	33	123	76	10	87	94	...	...	82	...	...
3	89	14	103	84	3	87	92	15	108	87	4	90	95	95	95	89	94	92
4	86	37	122	74	9	83	86	43	128	71	11	83	103	90	97	88	89	89
5	91	7	98	90	5	95	93	5	98	92	5	98	90	...	...	90	...	...
6	82	14	96	80	9	89	83	17	99	81	16	97	90	...	...	88	...	...
7	80	13	93	79	6	86	83	19	101	82	5	87	97	93	95	89	93	91
8	48	49	96	47	29	76	44	73	117	41	35	77	115	87	101	91	86	89
Average, % of known cement content	82	22	104	77	9	86	83	27	110	78	11	89	97	91	97	88	91	90
Average error, % of known cement content	...	...	8.1	...	...	13.8	...	...	11.0	...	...	10.8	7.1	8.8	3.5	11.6	9.5	9.8

tained in the hardened concrete, and, as mentioned previously, the method which results in the least contamination from soluble portions of the aggregate is to be preferred.

The presence of finely divided clay, silt, and pozzolans will vitiate the results based on a silica method alone. This is not generally true when calcium oxide is indicated as a basis of estimation. However, due to the widespread use of aggregates with high contents of acid-soluble calcium, a method based on calcium oxide determinations cannot be used in many instances.

ASTM Test for Cement Content of Hardened Portland Cement Concrete (C 85) is reasonably typical of most of the chemical methods and, therefore, is used as the basis of the description presented in this report. Several investigators have studied the ASTM method or modifications of it [*16–21*]. None of the procedures as reported are completely reliable and the investigators agree that unless a good deal is known about the aggregates the results will not give consistently accurate cement content values. Tables 4 and 5 are presented (adapted from the paper by Ford) to illustrate the variation in the results as determined by three modifications of ASTM Method C 85 – 42. The high degree of error shown in Table 5 using the silica method may be due to the solubility of the silica in the natural silica sand and gravel used as the aggregate in that work.

Ford points out that none of the methods may be depended upon to give results which deviate by less than 10 to 20 per cent from the known cement content for the eight samples of concrete and mortars tested. He also points out that the rich mixes usually give better values than lean mixtures and, in general, that values based on calcium oxide determinations show better results than methods based on soluble silica. As indicated previously, however, the calcium oxide method is also limited in usefulness and cannot be employed with limestone aggregate or with concrete that has been materially altered by leaching. For the most part, the values obtained using acid soluble silica techniques are found to be low. Calculations made of correlation coefficient and the variance analysis supported these general conclusions.

With the above observations in mind, the ASTM Subcommittee III-1 of Committee C-9 has investigated the limitations of Method C 85 – 54 and has developed several modifications directed to improving the reliability of the method. A more precise technique has been developed for leaching the sample with hydrochloric acid; the acid solution is heated to near boiling, digested for 10 min, and filtered through a Buchner type Coors porcelain funnel using two disks of Type 2 Class G filter paper. Hot NaOH solution (10 g per liter) is added to the acid residue; the resulting solution is heated to near boiling and digested for 15 min with occasional stirring before filtering.

As noted previously, consideration is also being given to a modified procedure for preparation of the test sample which involves decomposition of the concrete by heating with subsequent physical separation into two size fractions. The coarse aggregate is removed from the mix by separation of the dehydrated concrete over a No. 6 sieve. This coarse material is worked with a rubber tipped pestle to remove adhering cement and then washed with dilute acid, the resulting weight loss representing part of the cement in the dehydrated concrete. A sample of the fine aggregate used in the concrete mixture is obtained for analysis by screening a portion of the fraction passing the No. 6 sieve on a No. 50 sieve. The fine aggregate, the fine fraction of the concrete mixture (that is, the fraction passing the No. 6 sieve), and the cement used in the mixture (when available) are analyzed for soluble silica or calcium oxide content in accordance with the chemical procedures noted above. The decimal fraction by weight of cement present in the fraction passing the No. 6 sieve is determined from the equation

$$C_f = \frac{S_f - S_a}{S_c - S_a} \ldots\ldots\ldots (1)$$

where:

C_f = decimal fraction by weight of cement present in the fraction passing the No. 6 sieve,

S_f = weight percentage of soluble constituent (silica or calcium oxide) present in the fraction passing the No. 6 sieve,

S_c = weight percentage of soluble constituent (silica or calcium oxide) known or assumed to be present in the cement, and

S_a = weight percentage of soluble constituent (silica or calcium oxide) present in the aggregate in the fraction passing the No. 6 sieve.

This equation has been drafted from the following relationships:

$$S_f = C_f S_c + A_f S_a$$

$$C_f + A_f = 1.0$$

where:

A_f = decimal fraction by weight of aggregate present in the fraction passing the No. 6 sieve, and where C_f, S_f, S_c, and S_a are defined above.

The percentage by weight of cement in the sample of dehydrated concrete is then calculated by the equation

$$C_t = \frac{C_f W_f + W_L}{W_s} \times 100 \ldots (2)$$

where:

C_f is calculated from Eq 1,

C_t = weight percentage of cement in the oven-dried concrete,

W_f = weight of the fraction passing the No. 6 sieve,

W_L = weight lost during acid wash of the fraction retained on the No. 6 sieve, and

W_s = weight of the dehydrated sample.

Where this method is used, the need for correction factors related to the aggregate solubilities (as such) and the need for assumptions about proportioning of the aggregates are eliminated. Further, the solubility of the coarse aggregate is a matter of concern only as it relates to the solubility of the aggregate in the dilute hydrochloric acid used in the acid wash of the coarse aggregate. This soluble fraction is considered to be cement (only). Should some of the soluble fraction of the coarse aggregate be extracted during this wash, as could easily occur with some limestone aggregates, a small error would be introduced. When reasonable care is taken in the washing process the effect of this is insignificant in the over-all calculation. This method shows sufficient promise that it may eventually serve as the basis for an ASTM standard.

The ASTM subcommittee has also developed a method for calculating the the portland cement content on the basis of pounds of cement per cubic yard. A separate sample of the concrete, preferably at least three times in minimum dimension the maximum size of the aggregate, is wire brushed to remove loose material and foreign matter, dried to constant weight at 105 C, weighing (A) soaked in water for 1 hr, surface dried and weighed in air (B), then weighed in water (C). The reference unit weight to which the analytically determined cement content is referred is: $1686A/B - C$ = pounds oven-dry concrete per cubic yard.

TABLE 6—CEMENT CONTENT BY CHEMICAL METHOD (ASTM METHOD C 85 - 54[a]).

Laboratory No.	Series I Concretes Ground by Each Laboratory Separately								Series II All Concrete Samples Ground by One Laboratory			
	Correction for Aggregate Solubility Made[b]				Correction for Aggregate Solubility Not Made[c]				Correction for Aggregate Solubility Not Made[d]			
	423-lb Mix[e]		569-lb Mix[f]		423-lb Mix[e]		569-lb Mix[f]		423-lb Mix[e]		569-lb Mix[f]	
	SiO_2 Basis	CaO Basis	SiO_2 Basis	CaO Basis	SiO_2 Basis	CaO Basis	SiO_2 Basis	CaO Basis	SiO_2 Basis	CaO Basis	SiO_2 Basis	CaO Basis
1	9.8	9.2	15.3	12.4	13.0	9.6	18.4	12.8	12.0	9.7	13.5	12.9
2	9.1	10.1	13.2	13.5	11.9	10.1	16.0	13.5	11.4	9.4	13.1	12.1
3	7.9	8.6	13.0	12.1	10.2	8.9	13.9	12.4	12.2	9.6	14.2	12.3
4	7.5	8.8	11.6	12.1	9.8	9.0	13.9	12.4	11.3	9.3	13.4	12.1
5	11.4	11.8	14.3	16.0	14.5	11.8	17.2	16.0	11.3	9.5	12.9	12.2
6	10.0	9.2	11.8	12.9	13.3	9.6	15.5	13.2	12.9	10.6	15.2	13.3
Average determined cement content	9.28	9.62	13.20	13.17	12.12	9.83	15.82	13.38	11.85	9.68	13.72	12.48
Mean deviation (from determined average)	±1.12	±0.89	±1.07	±1.06	±1.48	±0.74	±1.38	±0.91	±0.52	±0.31	±0.66	±0.41

[a] Determined in accordance with the modifications of the chemical portion of the procedure outlined in text.

[b] Corrections based on assumed concrete mix proportions of 1 part cement, 2 parts fine aggregte, and 4 parts coarse aggregate by weight.

[c] Variable high per cent cement contents resulting from the SiO_2 method are caused by the slight solubility of the quartzite and silica sand which were used as the aggregate in this study.

[d] Determination of soluble constituents of the aggregates not included in the second series of tests.

[e] 423-lb cement (10.9 per cent by dry weight), 1480-lb fine aggregate, 1975-lb coarse aggregate, and 36.0-gal water.

[f] 569-lb cement (14.7 per cent by dry weight), 1360-lb fine aggregate, 1945-lb coarse aggregate, and 37.7-gal water.

Table 6 is a summary of data obtained from a cooperative test program carried out by Subcommittee III-1 of ASTM Committee C-9. Two series of tests were conducted, the first series of tests being run on samples of concrete which were sent intact to the various laboratories and the second series of tests being run on individual samples of the same concretes which had been prepared (ground) in one laboratory. In all cases each laboratory's sample consisted of one half of a standard concrete cylinder which had been sawed lengthwise.

The results of these tests indicated that sample preparation must be considered as one of the primary variables. Examination of the data as shown in the table indicates that the average deviations fall within ±1.5 per cent of the mean. The method based on calcium oxide determination is more consistent than that based on silica determination. In general, the agreement between laboratories is encouraging.

A procedure based on a combination of methods may produce more accurate results than have been realized by the use of the existing chemical procedures alone. To this end it would be suggested that the sample be initially analyzed by a microscopical method to determine the volumetric paste and aggregate content. Then, from a separate determination of specific gravity on the aggregates, the weight proportions of the aggregate per cubic yard in the concrete can be established. These proportions can then be used in making the calculations for the chemical method described in ASTM Method C 85 – 54. This procedure enables the analyst to make more precise corrections for soluble silica and calcium oxide in the aggregate, which is particularly important where the solubility of the aggregates is high.

SUMMARY

1. The determination of portland cement content of hardened concrete is influenced considerably by the materials used in the preparation of the concrete mixture. The presence of limestone, dolomite, slag, cinders, clay, pozzolans, and other materials that release calcium or silica seriously affect the usefulness of the presently employed chemical methods.

2. The type of aggregate dictates the particular chemical procedure that must be employed in making the determination, predominantly siliceous aggregates generally requiring a determination based on the calcium oxide content and calcareous aggregates requiring a determination based on the silicon dioxide content.

3. While adequate information on the separate ingredients of the mixture will generally improve the accuracy of the test, the chemical procedure has not yielded a high degree of accuracy. Results generally reported for ASTM Method C 85 – 54 vary from the known cement content by about 5 to 15 per cent.

4. ASTM Subcommittee III-1 of Committee C-9 has investigated the limitations of Method C 85 – 54 and is considering revision of the specification to include a new method of sample preparation, more precise chemical procedures, and modified calculations.

5. Recent investigations using physical techniques such as microscopical point-count analyses have given encouraging results. If the analysis is made over sufficient traverse length and area, results should come within 10 per cent of the theoretical cement content.

6. Where the solubilities of the aggregates used in the concrete mixture are large, a combination of physical and chemical analyses appears to be a more reliable means of making the determination than is the use of the chemical procedures outlined in ASTM Method

C 85 – 54 alone, in that the corrections for aggregate solubilities will be more accurate when the aggregate proportions have been carefully established.

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Special Categories

READY-MIXED CONCRETE

By Stanton Walker,[1] Honorary Member, ASTM

Ready-mixed concrete—defined as concrete sold as a finished product ready for use in a plastic and unhardened state—represents the largest category of portland cement concrete used in the United States. Approximately 150,000,000 cubic yards were produced in 1964. That accounts for about 60 per cent of all the cement sold in this country. The growth of the industry has been very rapid. In 1930 there were fewer than 200 companies producing not more than 5,000,000 cubic yards. The 150,000,000 cubic yards for 1964 come from some 5,000 to 6,000 companies.

There has been a similar dramatic development in equipment to produce ready-mixed concrete. Prior to about 1930, the typical central mixer had a capacity of 1 to 1½ cubic yards, although there were a few of 3 cubic yard capacity, and a few plants had more than one mixer. The concrete was transported in dump trucks, in special watertight open bodies, and, to a lesser extent, in the then recently developed agitators. The open trucks commonly had capacities of 1 to 2 cubic yards, and the agitators were only a little larger—about 2 to 3 cubic yards.

Today central mixers of 6 cubic yard capacity and larger are the general rule. Many have capacities of 9 to 10 cubic yards and more. The predominant current practice is to use a truck mixer for the delivery of the centrally mixed concrete. Open bodies or agitators not suitable as mixers are used principally under special circumstances—generally for a particular job.

By 1930, truck mixers had been developed. Capacities of 2 to 3 cubic yards were most common, with a few up to 5 cubic yards. The current standards of the Truck Mixer Manufacturers Bureau [*1*][2] include truck mixers from the minimum size of 5 cubic yards up to a maximum of 16 cubic yards. Corresponding agitator ratings are from 6½ up to 21¾ cubic yards—and provision is made for the acceptance as standard of larger units.

According to a 1963 study of the National Ready Mixed Concrete Assn., covering about 1500 companies, 78 per cent of the volume of ready-mixed concrete came from the transit- or truck-mixing operation. The companies reporting operated 3282 plants of which 68 per cent were batching and 32 per cent central mixing plants.

Batching plants have met the needs of larger mixers and agitators and have made much progress in keeping up with the demands for more accurate control which has accompanied the greater use of portland cement concrete, whether job-site mixed or delivered ready mixed. Standards of the Concrete Plant Manu-

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

facturers Bureau [*2*] provide specifications covering performance and capacities for all elements of a concrete batching system in five categories: (*a*) manual, (*b*) partially automatic, (*c*) semiautomatic, (*d*) automatic, and (*e*) automatic with mix selection. Information from the Bureau shows that of the plants sold during the past two or three years, 65 per cent of the aggregate batchers, 78 per cent of the cement batchers, and 55 per cent of those used for measuring mixing water fall in one of the four automatic categories.

Controlling the quality of ready-mixed concrete presents only a few problems, but highly important ones, not found in other concrete production operations. Of these, probably the most significant is that the responsibilities for the final quality of the concrete is divided. The producer delivers the concrete to the user who places it in the work and gives it whatever subsequent treatment it receives. Therefore, measures of the acceptability of the plastic concrete at the point where the responsibility for its handling passes from the producer to the user are of particular importance because of the contractual relations involved.

Most problems of controlling the uniformity of quality of ready-mixed concrete revolve about the measurement of mixing water and the maintenance of the desired consistency. Compensation must be made for the surface moisture on the aggregates. Mixed concrete stiffens and, therefore, the water demand is affected by length of haul, time and rate of mixing, time required to discharge the batch, temperature, characteristics of the cement, and so on. The large sizes of batches contribute to the element of time.

The description and history of the development of ready-mixed concrete cannot be covered adequately here. However, what little has been said emphasizes the need for continuous attention to specifications and test methods pertinent to the control of quality of ready-mixed concrete. For those who wish to pursue the subject, helpful references are included in the appended list. See in particular [*3–9*].

SPECIFICATIONS

Most specifications for ready-mixed concrete concern themselves principally with the bases for its acceptance by the purchaser and with features of the operation peculiar to its production and delivery. Such specifications are supplementary to a general specification covering the complete project. The ASTM Standard Specifications for Ready Mixed Concrete (C 94) [*10*] recognize this principle; while it includes requirements for quality of materials and proportions, it provides that the applicable general specifications shall govern, unless otherwise specified by the purchaser.

This discussion of specifications is restricted to the supplementary features required to define the responsibilities of the ready-mixed concrete producer. It should be clear that these must be based on the quality of the concrete as delivered; the producer has no jurisdiction over the treatment given it after delivery, a significant factor in ultimate quality.

Bases for Acceptance:

Specifications for ready-mixed concrete fall in three broad categories: (1) the prescription type; (2) the performance type; and (3) a combination of the two, in which the purchaser specifies both performance and limitations on prescription.

In the prescription approach, the producer has met the requirements if he incorporates in the batch the quantities of materials called for and complies with limitations on such factors as slump, capacities of truck mixers and agitators, rate and number of revolutions of mixing, elapsed time between adding the water and completing the discharge of the batch, and uniformity of mixed concrete. Complete assurance that the prescription has been met requires that all operations be conducted under the supervision of a qualified representative of the purchaser. That practice is economically realistic only when considerable quantities of concrete are being furnished continuously during a given period.

For the average circumstance of intermittent delivery of relatively small volumes of concrete, certification on the part of a responsible producer that he has complied with the specifications should afford adequate protection. The details of the items to be certified should be clearly stated. The purchaser should make a sufficient number of inspections and tests to determine that batching, mixing, and transportation procedures are consistent with the requirements to be met. Automation has done much to surround the prescription specification, however arrived at, with adequate safeguards.

In the performance approach, the purchaser customarily specifies only the necessary characteristics of the materials, the required strength, the range in slump, and, for air-entrained concrete, the range in air content. The producer is responsible for selecting suitable materials and proportions. This type of specification, as well as the combination of prescription and performance, presents some difficult problems of administration. Strength is almost always the ruling criterion. Results of strength tests are affected by numerous factors, many of which may be completely independent of the quality of the concrete. The results of strength tests are not known until the concrete is in the forms and hardened.

A major area of misunderstanding has to do with what constitutes a suitable strength test. Strength test results, apart from actual strength, may be seriously affected by sampling and molding procedures, kinds of molds, and, in general, handling and protection on the job, en route to the laboratory and in the laboratory. When measured strength is a condition of acceptance, the tests to be significant must be made under standard conditions including, in particular, the protection of the specimens from the time they are molded until they are tested. Enough tests must be made to present a dependable picture. Even with the tests carried out with meticulous concern for good practice, allowance should be made for variations which are inherent in the whole procedure. Various criteria have been considered to take these variations into account.

Criteria for Strength:

ASTM Specification C 94 requires that "the average of all of the strength tests representing each class of concrete, as well as the average of any five consecutive tests for each class of concrete, shall be equal to or greater than the specified strength, and not more than one test in ten shall have an average value less than 90 per cent of the specified strength." The Building Code Requirements for Reinforced Concrete of the American Concrete Inst. (ACI 318) [*11*] specify two criteria. For concrete used in structures for which the analysis is based on working stress design, it is required that "the average of any five consecutive strength tests of laboratory-cured specimens representing each class of concrete shall be equal to or greater than the specified strength, f'_c, and not more than 20 per cent of the strength tests shall have values less than the specified strength." For ultimate strength design and for prestressed structures, the limitations are more rigid and require the average of any three consecutive tests to be equal to or greater than the specified strength and not more than 10 per cent to have values less than the specified strength.

These provisions are feasible of administration for any project where a sufficient volume of concrete is consumed to permit securing the minimum number of specimens to make the "10 per cent" or "20 per cent" meaningful. One approach to minimize that problem is to specify that the average strength of a stated minimum number of tests shall govern, with no limitations on the minimum for an individual test. For example:

TABLE 1—ESTIMATE OF COEFFICIENTS OF VARIATION FOR DIFFERENT QUALITIES OF CONTROL.

Coefficient of Variation, per cent	Quality of Control
Under 5.......	attainable only in laboratory
5 to 10.......	excellent for field
10 to 15.......	good for field
15 to 20.......	fair for field
Over 20.......	poor for field

> To conform to the requirements of these specifications, the average strength of any —— consecutive tests shall be equal to or greater than the strength specified (f'_c).

It is believed that the insertion of the number 5 in the blank space makes for a practical and adequately protective provision. The insertion of the number 3 would represent a more rigid requirement which it is believed could be met with reasonable economy and which should eliminate substantially any objection to the absence of an absolute minimum for a single test.

Average Versus Specified Strength:

Variations inherent in the strength test, independent of the quality of the concrete, are large enough to be significant. That is not surprising when consideration is given to: the small size of specimen in relation to the size of the larger particles in the concrete, the difficulty of securing a representative sample consisting of only about one half of one per cent of the batch of a material so heterogeneous as concrete, and the effects of other variables of handling and protection referred to previously.

The variables affecting the uniformity of concrete are largely random, and, therefore, the normal probability equation gives a good measure of the distribution of strength tests [*12*,*13*]. The standard deviation, or the coefficient of variation, provides a useful tool for estimating an average strength needed to afford good assurance of meeting strength limitations such as those just described. Table 1 [*12*,*13*] gives a good estimate of strength variations, expressed in terms of coefficient of variation, for various qualities of control. Table 2 translates this information to show the amount of over-design required to limit chances of failure to 1 in 10 and 1 in 100 for given qualities of control.

TABLE 2—REQUIRED OVER-DESIGN FOR DIFFERENT COEFFICIENTS OF VARIATION.

No. of Consecutive Tests Included in Average	Average Coefficient of Variation of Individual Tests		
	10	15	20
OVER-DESIGN FOR 1 IN 10 CHANCES OF FAILURE			
1.......	115	124	135
2.......	110	116	123
3.......	108	113	118
4.......	107	111	115
5......	106	110	113
OVER-DESIGN FOR 1 IN 100 CHANCES OF FAILURE			
1......	130	154	188
2......	120	133	153
3......	116	126	137
4......	113	121	130
5......	112	118	126

This suggests the need for an advisory

note to accompany a strength specification. Some such wording as the following [*14*] has received considerable discussion:

> NOTE—Because of variations inherent in concrete materials, production, sampling, and testing, the average strength of the concrete must be significantly higher than the specified strength if the requirements specified are to be met. The amount by which the average strength should exceed the specified strength will depend upon the uniformity of the concrete and upon the uniformity of the sampling and testing procedures. The coefficient of variation of test results, calculated from as many pertinent tests as are available, but not fewer than five, affords a good basis for estimating the average strength needed by means of the following formula:
>
> $$f_{cr} = \frac{f'_c}{1 - V}.$$
>
> where:
>
> f_{cr} = needed average strength of concrete,
> f'_c = specified strength of concrete, and
> V = coefficient of variation expressed as a decimal.

That note means approximately that if an average strength of f_{cr} is maintained, about 4 per cent of the averages of three consecutive tests will fall below f'_c; for five consecutive tests, a little more than 1 per cent of the averages will fall below f'_c. Further, it is suggested that concrete test data depart from the normal probability curve enough so that the "4 per cent" and "little more than 1 per cent" may be conservative.

Penalties:

The assessment of realistic penalties for failure to meet strength specifications must depend greatly on engineering judgment. Failures are not black or white. If a strength of 5000 psi is specified, a group of tests averaging 4900 psi constitutes a failure, but not one of the same degree as if the attained strength were 25000 psi. The penalties on everyone concerned, both producer and purchaser, would be unrealistically great if the 4900 psi concrete were removed from the structure; but failures by even as little as 100 psi should not be condoned to the point of being considered generally acceptable.

ASTM Specification C 94 has no adequately spelled out penalty clause. It has a section entitled "Failure to Meet Strength Requirements" which reads, in part, as follows:

> In the event that concrete . . . fails to meet the strength requirements of these specifications, the manufacturer of the ready-mixed concrete and the purchaser shall confer to determine whether agreement can be reached as to what adjustment, if any, shall be made. If an agreement on a mutually satisfactory adjustment cannot be reached by the manufacturer and the purchaser, a decision shall be made by a panel of three qualified engineers, one of whom shall be designated by the purchaser, one by the manufacturer, and the third chosen by these two members of the panel. The question of responsibility for the cost of such arbitration shall be determined by the panel. Its decision shall be binding, except as modified by a court decision.

Various attempts have been made to write "money" penalty clauses, but they have not received significant acceptance. They are difficult to interpret. For example, to what volume of concrete does the penalty apply? Also, such clauses generally have been considered either too innocuous or too punitive.

The solution to deficiencies in measured strength has generally been arrived at by core tests or load tests with some sort of an adjustment being made based on engineering judgment. Rarely is it economically feasible to remove a substantial portion of the structure. Generally, it is feasible to strengthen it so as to leave no doubt of its safety so far as strength is concerned. On the other hand, the problem is complicated by the fact that deficiencies in strength which may not endanger "life and limb" may very well make for significant reductions in durability.

Some such basis for a penalty clause as follows may be worthy of consideration [*14*]:

1. A strength test representing no more than an agreed upon volume of concrete.
2. Graduated penalties in reduced price of the concrete as delivered to the job up to 100 per cent at some agreed upon deficiency in strength—perhaps something of the order of 20 per cent.
3. Beyond this 100 per cent point, agreement along some such lines as the ASTM arbitration clause with emphasis on necessary strengthening or protection of the concrete in question.

OPERATING PROCEDURES

Operating procedures include everything having to do with the manufacture and delivery of the concrete. They start with the receipt and handling of the solid ingredients into storage and continue through all of the many steps necessary for putting the concrete in the hands of the purchaser. It is not feasible for any specification to cover the details of such procedures, since they vary so much for different kinds of operations.

The principal essential features of operations covered by typical specifications are:

1. Accuracy of measurement of materials, including requirements for the batching plant in which they are measured.
2. Mixing and delivery, including amount and rate of mixing, time between mixing and discharge, requirements for uniformity of mixing and discharge, and, sometimes, limitations on temperature in hot and cold weather.
3. Requirements for the performance of agitators and mixers, including stationary mixers. At this time, these generally consist of a statement that uniform and homogeneous concrete shall be produced and accompanied, sometimes, by limitations on range in slump. More detailed requirements, discussed later, are being considered for specifications now in process.
4. Inspection and methods of sampling and testing.

The appended list of references affords comprehensive information dealing with the many facets of operating procedures. Particular attention is directed to the following:

1. Standard Specifications for Ready-Mixed Concrete (ASTM C 94) [*10*].
2. Recommended Practice for Measuring, Mixing, and Placing Concrete (ACI 614) [*15*].
3. Control of Quality of Ready-Mixed Concrete (NRMCA Publication 44, October, 1962) [*16*].
4. Standards for Operation of Truck Mixers and Agitators (NRMCA Publication 73, 1957) [*17*].
5. Recommended Practice for Measuring the Uniformity of Concrete Produced in Truck Mixers (NRMCA Publication 105, 1962) [*18*].
6. Recommended Guide Specifications Covering Plant and Accessory Equipment for Ready-Mixed Concrete in Construction for Highways (NRMCA Publication 109, 1962) [*19*].
7. Standard Practice for Concrete (Office Chief of Engineers, Department of the Army, 1963) [*20*].
8. Standard Guide Specifications for Concrete (Office Chief of Engineers, Department of the Army, 1963) [*21*].
9. Manual of Concrete Inspection (ACI 611) [*22*].

There follows comment on operating procedures, including many not covered by specifications.

Measuring Materials:

The accuracy with which materials should be measured is a part of any complete specification. Common requirements [*19*] are:

Cement and other cementitious materials	±1 per cent
Aggregates	±2 per cent
Water	±1 per cent
Admixtures	±3 per cent

ASTM Specification C 94 [*10*] modifies the limitations on accuracy for weighing aggregates by specifying the ±2 per cent for individual aggregates but requiring the total weight to be within ±1 per cent.

Suitable scales regularly used in the ready-mixed concrete operation are fully capable of weighing within these limitations. However, there are other factors affecting accuracy, prominent among which, particularly for automated equipment, is the amount of material in free fall between the bin gate and weighing hopper after the bin gate is closed. It is desirable, therefore, that the weight of material in the weighing hopper be checked from time to time by manual operation of the equipment. Also, of course, the calibration of scales should be checked at suitable intervals.

The limitation of ±1 per cent on the accuracy with which water is measured appears to be unrealistic. So far as water measured directly into the batch is concerned, this accuracy is readily attainable. However, when moist aggregates are used, such a limitation presents difficulties. An error in knowledge of the moisture content of only $\frac{1}{2}$ of 1 per cent in the fine aggregate and $\frac{1}{4}$ of 1 per cent in the coarse aggregate, both in the same direction, amounts to about 4 per cent of the total mixing water. For air-entrained concrete, small differences in the air content affect the water demand [*23*].

There are, of course, other factors involved in determining how much water should be added to the batch. The slump of the concrete is of significance at the time the concrete is discharged from the mixer or agitator. This means that water demand may vary with circumstances—principally time and temperature, discussed earlier.

The preceding suggests that specifications for mixing water should place principal emphasis on the maximum quantity permissive, with the amount for a given batch being that, within this limit, required to produce the desired consistency. This procedure makes necessary the measurement of the actual quantity added and that introduced as surface moisture of the aggregates to be assured that the water demand does not exceed the maximum specified. It also indicates the high desirability of a device for measuring consistency of the concrete in the mixer.

Measuring Moisture in Aggregates:

A large proportion of modern ready-mixed concrete operations make use of one of the several electrical moisture meters available on the market. For the most part, these are applicable to fine aggregate only. At least one device has been used with the finer sizes of coarse aggregate as well as with the fine aggregate. These meters measure the electrical resistance of the moist aggregate which varies with moisture content. Their accuracy depends on the care with which calibration is maintained [*24,25*]. Checks against determination by drying should be made at frequent intervals. Such meters work best with concrete sands having moisture contents of less than about 7 or 8 per cent, the upper limit depending on the sand grading as it affects water-carrying capacity. For sand wet enough to drain readily, material below the electrodes of the meter will contain more water than that in contact with the probes which determine the resistance [*25*], a condition particularly prevalent at the start of daily operations.

One resistance meter on the market "integrates" continuous moisture readings taken throughout the discharge of the aggregates into the batcher. It also has been adapted to automated equipment so as to correct weights of aggregates and added mixing water for aggregate surface moisture. Literature describing moisture meters is available from their manufacturers. A current list of manufacturers can be secured from a number of organizations, including the Portland Cement Assn. and the National Ready Mixed Concrete Assn.

Experimental work has been done on instruments depending on more complex electrical characteristics than resistance and, also, on nuclear radiation. One of these is described as a "highly efficient scintillation counting process," utilizing "neutron-hydrogen interaction." It is understood that one important deficiency of the radiation devices, aside from that involving the maintenance and handling of a nuclear energy source, has to do with the time involved in making the determination. One such device, for "scanning" the material in the weighing hopper and used experimentally in a ready-mixed concrete plant, required a minimum of 90 sec to complete the measurement.

The feasibility of using simple drying procedures or a displacement procedure, such as described in ASTM Test for Surface Moisture in Fine Aggregate (C 70), should not be overlooked when conditions are favorable. Their satisfactory use demands aggregates be stored in such a manner that changes in moisture content are gradual enough so that measurements at intervals of 1 to 2 hr or more will give an adequate picture.

Measuring Consistency:

Recognition of the importance of consistency in controlling the quantity of mixing water has led to a number of efforts to measure that characteristic of concrete in the mixer drum [*26–29*]. For the most part, these attempts have been directed to stationary mixers and have included: (1) measurement of resistance to movement of a device located inside the mixer drum; (2) measurement, along the horizontal, of the location of the center of gravity of the batch in a tilting mixer; and (3) power required to rotate the mixer drum or blade.

For the first approach, certain devices gave a good measure of consistency but were found to be impractical because of the severe wear on them. The center of gravity approach left much to be desired as to accuracy and was limited in application. The power input approach has been developed to the point where it provides a helpful tool for the control of consistency—when mixers are electrically driven and if a special wattmeter or ammeter is used.

For truck mixers using gasoline engines, considerable attention has been paid to the determination of power input through measurement of manifold pressures. This has been more successful for truck mixers with a separate engine drive for the mixer drum; however, such devices have also been used on truck mixers with power take-off. They are most effective when power is being consumed only for the revolution of the mixer drum, that is, with the truck not in motion. Consideration has been given to measurement of power by pressure in hydraulic driving mechanisms, but these have not been developed for that purpose.

Mixing and Delivery:

Because concrete stiffens with time and the rate of stiffening is affected by temperature and work, the conditions for mixing and delivery need to be stated in considerable detail. The requirements and recommendations of ASTM Specification C 94 are used widely as a guide. A summary of these will serve to illustrate accepted practice.

The time between the introduction of the mixing water to cement and aggregates, or the cement to the aggregates, and the completion of the discharge of the batch is limited to $1\frac{1}{2}$ hr or 300 revolutions of the drum, whichever comes first, unless a longer time is specifically authorized. Also, the standard points out, a lesser time may be required in hot weather or under conditions contributing to quick stiffening of the concrete. A further limitation on the time element is that the mixing operation shall begin within 30 min "after the cement has been intermingled with the aggregates."

Specifications other than ASTM are frequently less liberal on limitations on time. While about one third of the state highway departments provide for times up to 90 min, this limitation is variously modified—as, for example, to 60 min "for hot weather," "for temperatures greater than 85 F," and so on. The more common upper limit specified is 60 min, and there are a number at 45 min, with some of these being modified—as, for example, to 30 min for Type III cement [*30*].

For truck mixers the only significant measure of amount of mixing is number of revolutions; time is not an accurate indication because the rate of revolution may vary over a wide range. Rate of mixing is significant, and the minimum mixing speed is conventionally specified as 4 rpm. The maximum rate is generally limited to a peripheral speed of 225 ft/min—for the typical mixer about 11 rpm.

A minimum number of revolutions at mixing speed is needed, and this is generally specified as 70. It has been considered desirable to establish a maximum number of revolutions at mixing speed to limit the amount of work done on the concrete—that is, the amount of grinding and heat generation. This upper limit is usually 100; all additional revolutions are required to be at agitating speed, specified as from 2 to 6 rpm. In comparatively recent years, an additional limitation has been added—a maximum limit on total revolutions whether at mixing speed or at agitating speed. The current ASTM limit is 300. It would seem reasonable to simplify these limitations by stating the minimum number of revolutions required at mixing speed with an over-all limit on revolutions at any speed. For example, a minimum of 70 revolutions at more than 4 rpm and less than, say, 11 rpm might be specified together with an over-all limit at any rate.

The requirements for numbers of revolutions in ASTM Specification C 94 need to be stated more clearly. Truck mixers are loaded, and mixing water is injected with drums revolving. The point from which the number of revolutions should be counted is not clearly defined. The Standard of the American Association of State Highway Officials, in most respects identical with ASTM Specification C 94, clarifies this point by stating that "all materials, including mixing water, shall be in the mixer drum before actuating the revolution counter."

The intent of this is clear, but it does not cover the common, almost routine, practice of the truck mixer arriving at the job with the concrete mixed to a plastic condition, but requiring the addition of water to increase the consistency to the condition needed for placement. The reason for this practice is clear; in the interest of accuracy, it leaves until the last minute the control of the final consistency of the concrete.

On the other hand, this practice has disadvantages, principal among which is the time required to thoroughly incorporate the added water into the batch.

So far as actual needs are concerned, limitations on time can be quite flexible. Longer periods can be tolerated for:

(*a*) cool weather than for hot weather,

(*b*) lesser amounts of work due to mixing and agitation (that is, lower number of revolutions) than for larger amounts, and

(*c*) hard aggregates than for soft, because of less grinding.

A good measure of permissive time, under varied circumstances, is mixing water demand. Any combination of time, work, and temperature which does not result in the need to increase mixing water beyond the specified limit generally should be permissive.

Adjustment of Consistency:

Whenever it is at all practicable, mixing water should be completely controlled at the batching plant. Nevertheless, however meticulous the control may be, either for central mixing or truck mixing, the need for adjustment of consistency at the job frequently presents itself, and water must be added or a perfectly good batch of concrete rejected. The problem here is to be assured that the added water is thoroughly incorporated in the concrete. This requires time and a minimum of 20 to 30 revolutions of the drum at mixing speed; and it also requires an adequate water injection system on the truck mixer.

Wash Water:

Water for washing the interior of the mixer drum after the concrete has been discharged commonly is carried on both agitators and mixers. If properly handled, this should cause no difficulties. Wash water is needed if the agitator or mixer is to be kept clean without undue expenditure of time. In the case of an agitator, the wash water should be completely discharged before the next batch is loaded, unless the quantity used is small enough as to have no significant effect on the characteristics of the concrete. In the case of truck mixers it is common practice to retain the wash water in the drum for reuse as a part of the mixing water for the succeeding batch, but this should be done only under careful supervision. There should be no objection to this practice if the wash water is free of foreign matter, such as hardened, set lumps of concrete broken free in the cleaning process—and *if it is accurately measured.*

Discharge Control:

All of the advantages to be gained from meticulous control of the operation prior to discharge may be thrown away by careless or improper discharge methods. Truck mixers of the revolving drum type have two, and in some three, mechanical features which combine to control the rate of discharge. All are equipped to permit reversal of the drum and variable drum speeds. Horizontal axis mixers and a limited and decreasing number of inclined axis mixers also have a variable discharge opening.

The three mechanisms—reversing, speed control, and discharge opening—are used separately or in combinations to control the rate at which the concrete is discharged. Familiarity with proper control of these features is necessary to prevent segregation and represents an operation which cannot be described successfully in specifications. Perfectly mixed concrete in the drum may be "unmixed" by improper discharge control. Segregation, due entirely to this cause, has often been attributed erroneously to defective mixing action.

Unrestricted discharge at full drum speed, with a full discharge opening, is desirable whenever the concrete can be disposed of at the maximum discharge rate—about 20 sec/yard^3 for typical truck mixers and concrete of the type most used in construction, for example, workable concrete made with aggregate having a maximum size up to about 2 in. and having a slump within the range of 3 to 5 in. Drum manipulation (direction and speed) is the best method for controlling discharge into wheelbarrows, buggies, or buckets.

TABLE 3—PERMISSIVE RANGE BETWEEN TWO SAMPLES.

Test	Stationary Mixer (Ref. *10*)	Truck Mixers		
		Ref. *18*	Ref. *19*	Author
Slump, in.	¾ or 25%[a]	2[b]	¾ or 25%[a]	1 or 25%[a]
Coarse aggregate[c]	6	5	5	5
Compressive strength, psi	7.5%[d]	10%[d]	7.5%[d]	7.5%[d]
Unit weight, pcf				
Concrete	...	1.5	...	...
Concrete (air free)	1	...	...	...
Mortar (air free)	0.8%	2	1	1½
Cement content	7%[e]	...	...	...

[a] ¾ in. (or 1 in.) or 25 per cent of average of two samples, whichever is greater.

[b] 1½ in. if determinations average of two tests.

[c] Materials coarser than No. 4 sieve, per cent by weight of concrete.

[d] Per cent of average strength for both samples at each test age; 7-day tests only specified for Ref. *10;* others imply 7 or 28 days or both.

[e] Alternate 1: alternate 2 is unit weight of air-free mortar.

Flat chutes should be avoided unless the concrete is helped along by means of shovels or hoes. Concrete made wet enough to flow of its own accord down a flat chute is not only wasteful of strength and cement, but it tends to segregate. The use of long chutes is to be avoided wherever possible. In discharging concrete from a chute or a spout, the discharge end should be near the surface of the concrete and should be swung back and forth to avoid coning and consequent segregation. A baffle at the end of the chute is frequently helpful in preventing segregation.

Measuring Uniformity:

The uniformity of the fresh concrete is the most direct measure of adequacy of mixing and of the satisfactoriness of discharge and other handling procedures. Much attention has been directed to the development of procedures and criteria for evaluating uniformity.

ASTM Specification C 94 has recently been revised to include bases for determining the mixing time for stationary mixers. The common limitation of 1 min for mixers of capacities of one cubic yard or less, with an additional "15 sec for each cubic yard or fraction thereof of additional capacity" is specified "where no mixer performance tests are made." But "when mixer performance tests have been made on given concrete mixtures," the mixing time may be reduced to the point where satisfactory mixing, as defined in the specifications, is accomplished—or it may be increased if found inadequate.

The criteria are permissive differences in test results for two samples taken from the mixer in a specified manner [*10*]. An approach of this type has been proposed as a basis for measuring the mixing efficiency of truck mixers, or, for that matter, any mixer [*18,19,31*]. Significant criteria and permissive ranges included in these proposals, together with modifications suggested by the author based

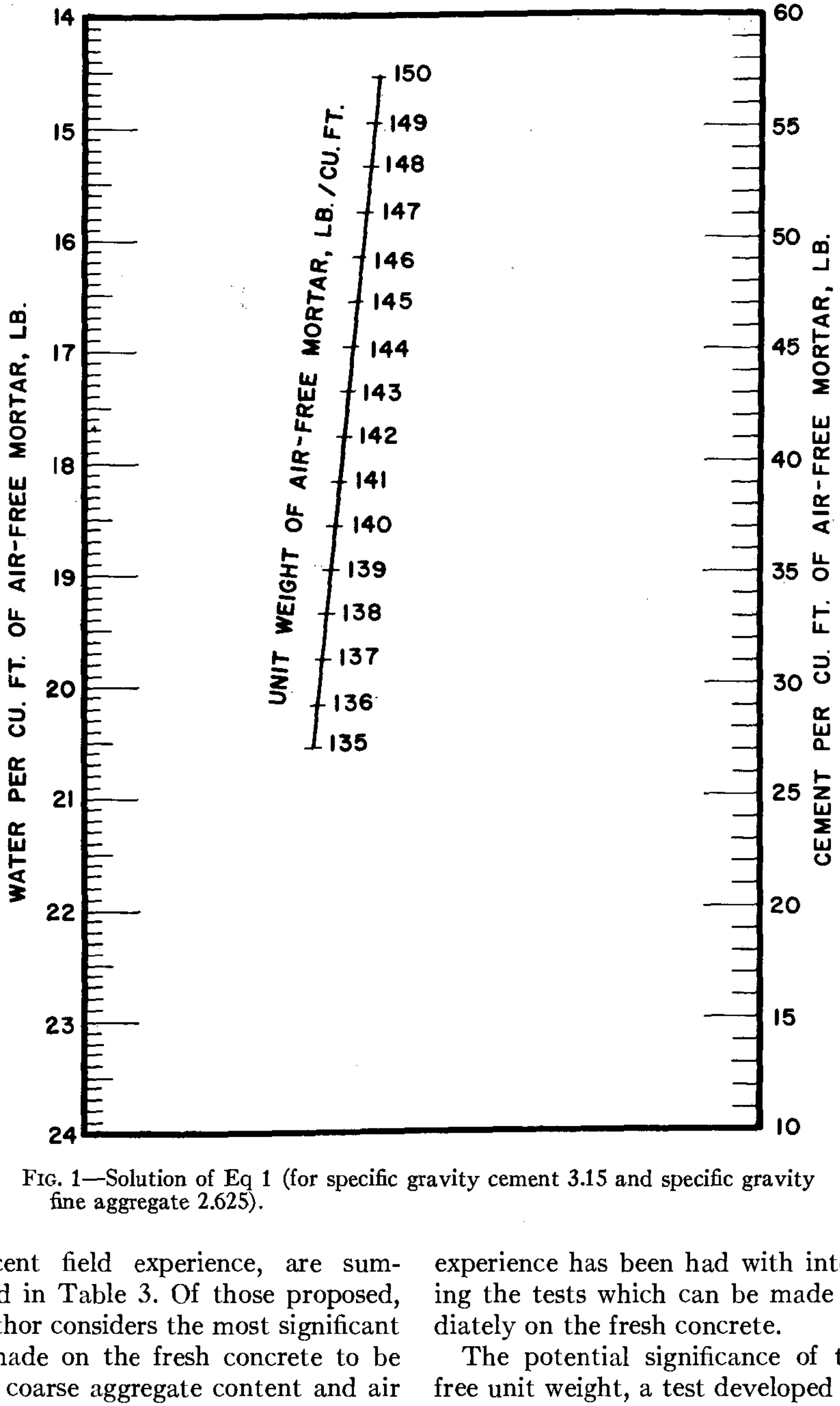

FIG. 1—Solution of Eq 1 (for specific gravity cement 3.15 and specific gravity fine aggregate 2.625).

on recent field experience, are summarized in Table 3. Of those proposed, the author considers the most significant tests made on the fresh concrete to be slump, coarse aggregate content and air free unit weight of mortar [*32*]. Strength tests at 7 or 28 days, or perhaps as early as 3 days, are considered a necessary supplement—at least until further experience has been had with interpreting the tests which can be made immediately on the fresh concrete.

The potential significance of the air free unit weight, a test developed by the Bureau of Reclamation as a measure of mixing efficiency [*32*], should be emphasized. It reflects water-cement ratio and, if determinations are adequately refined, which is not yet so, the cement content can be calculated for known water content—and vice versa. For a given amount of water per unit volume of air-free mortar, a change in unit weight of 1 lb/ft^3 is caused by about 6 lb of cement—which, for typical concrete, translates to about 80 to 90 lb/yard^3 of concrete. Similarly for a given amount of cement per unit volume of air-free mortar, a change in unit weight of 1 lb/ft^3 is caused by 0.6 lb of water—or, for typical concrete, about 9 lb/yard^3. For constant water-cement ratio, a change of 1 lb/ft^3 in the unit weight of air-free mortar represents a change in water content of about 0.8 lb (12 lb/yard^3) and in cement content of about 1.6 lb (24 lb/yard^3). It can be shown that the following general relationship exists:

$$C = \frac{1}{1 - G_a/G_c} \cdot [U + (G_a - 1)W - 62.4G_a] \quad (1)$$

where:

C = lb of cement/ft^3 of air-free mortar,

U = unit weight of air-free mortar lb/ft^3,

W = lb of water/ft^3 of air-free mortar, and

G_a and G_c = specific gravities of aggregate and cement, respectively.

Figure 1 is based on the relationship shown in Eq 1 assuming the specific gravity of the cement to be 3.15 and that of the fine aggregate 2.625.

Hot and Cold Weather Concrete:

Specifications recognize that the production of ready-mixed concrete is a year-round operation. Both hot and cold weather require special precautions if high quality is to be maintained, and the problems of hot weather are, in most respects, more difficult than those of cold weather.

As to hot weather, ASTM Specification C 94 contains only the admonishment that difficulties may be encountered with concrete at temperatures approaching 90 F, and it also states that "every effort shall be made to maintain temperature ... as low as possible." Suggestions for implementing these admonitions are to be found in the "Recommended Practice for Hot Weather Concreting," ACI Committee 605 [*33*]. See also "Cooling Ready Mixed Concrete" [*30*], a publication of the National Ready Mixed Concrete Assn.

ASTM Specification C 94 is more specific in its limitations on concrete in cold weather than in hot. It specifies minimum concrete temperatures ranging from 50 to 70 F depending upon the air temperature and the type of work (principally thickness of section) in which the concrete is to be used. It further requires that at no time during the production or transportation shall the maximum temperature of the concrete exceed 90 F. For more detailed information see "Recommended Practice for Winter Concrete," ACI 604 [*34*]; "Cold Weather Ready Mixed Concrete," NRMCA Publication No. 34 [*35*]; and "Control of Quality of Ready Mixed Concrete," NRMCA Publication No. 44 [*16*].

SAMPLING AND TESTING

Sampling:

The importance of sampling is apparent. If a sample is truly representative and if test specimens are made and handled properly, then the test results may be considered to reflect the quality of the concrete. On the other hand, samples which are carelessly taken cannot yield significant results no matter how well the tests are made. The exercise of informed judgment in selecting samples is indispensable.

ASTM Method of Sampling Fresh Concrete (C 172) outlines procedures for taking samples from stationary and paving mixers, truck mixers and agitators, and dump trucks or other types of open-top containers. The sample is required to consist of portions from different points in the batch. Under any circumstances the composite sample is to be remixed with a shovel the minimum amount to insure uniformity and protected from sunlight and wind during the period between taking and using, which is required not to exceed 15 min. If the sample is to be used for strength tests, it shall consist of not less than 1 cubic ft; smaller samples are sanctioned for routine slump tests.

The method is specific in one important and often neglected practice: "The sample shall be transported to the place where test specimens are to be molded or where the test is to be made, and shall be remixed with a shovel the minimum amount to ensure uniformity." Often the strength specimens are molded at some place immediately adjacent to the place where the concrete is being discharged and then transported to some out-of-the-way place for storage while they are still in a plastic state. Such a procedure cannot improve the strength of a properly molded cylinder and can very easily harm it.

ASTM Method C 172 makes no provision for securing samples of ready-mixed concrete from the forms. This is proper since the function of the ready-mixed concrete producer is to furnish mixed concrete to the purchaser at the job site. In his regular capacity he has no control after the concrete is discharged from the transportation vehicle.

Slump Test:

The first test commonly made on a sample of freshly-mixed concrete is for slump. In spite of its apparent lack of refinement, slump is a good measure of consistency and, consequently, for known materials, a good indication of the water content of the batch. In ASTM Specification C 94 the slump test is used not only as an acceptance test but also as a measure of the thoroughness and uniformity of mixing.

The slump test method is described in ASTM Test for Slump of Portland Cement Concrete (C 143). It says nothing of tolerances, but these are covered in some detail in Specification C 94. Section 5 reads:

> When the specified slump is 3 in. or less the tolerance shall be ±½ in. When the specified slump is greater than 3 in., the tolerance shall be ±1 in.

Specification C 94 also provides, in Section 13, that if the measured slump falls outside the limits specified, a check test shall be made. It is only after the second failure that the concrete is considered to have failed to meet the requirements of the specifications. This safeguard is in recognition of the sensitivity of the test to relatively minor departures from specified procedures, more likely in the field than in the laboratory.

A convenient supplementary test for consistency of concrete, but not a substitute for the slump test, is provided by ASTM Test for Ball Penetration in Fresh Portland Cement Concrete (C 360). It has the advantage over the slump test of not involving the molding of a specimen and of permitting the testing of concrete in a wheelbarrow or other container, or even the forms. The test can be made quickly and, thus, provides a convenient means for the routine checking of consistency for control purposes.

The results cannot be translated accurately into terms of slump and should not be used as a basis for acceptance or rejection of the concrete on the basis of slump requirements. The reliability of the penetration test is less for low slumps, large maximum sizes of aggregates, and harsh mixes than it is for workable mixes with aggregates of maximum sizes of 2 in. or less.

Compression Tests:

ASTM Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31 – 62 T) outlines the conditions for securing and preparing specimens ready for test. ASTM Test for Compressive Strength of Molded Concrete Cylinders (C 39) describes how they shall be tested. ASTM Specification C 94 requires, when strength is used as a basis for acceptance, that "tests shall be made frequently by the purchaser and, in general, not less frequently than one strength test for each 50 loads of each class of concrete, except that in no case shall a given class of concrete be represented by less than three tests." A single strength test is defined as consisting of tests of three standard test specimens. Provision is made for discarding defective specimens.

The most important and probably most often neglected provisions of ASTM Method C 31 are those pertaining to curing. Violation of these provisions will, almost without exception, lead to reduced strengths and, therefore, must not be tolerated. For instance, cylinders left unprotected on the job over the weekend can very easily lose 10 to 25 per cent of their potential 28-day strength [*36*]. Specimens should be stored in insulated storage boxes, or other suitable means should be used to obtain the temperature conditions required.

Specimens stored under damp burlap or sand usually will have a lower temperature than the surrounding atmosphere because of evaporation; this should be taken into account in maintaining the temperature of specimens within the limits of the standard. When specimens are transported from the field to the laboratory they should be securely packed to avoid damage.

Core Tests:

When tests of molded cylinders fail to meet strength requirements, it is common practice to test cores taken from the hardened concrete. It is obvious from earlier discussion that such tests may be highly prejudicial to the concrete producer because of the sins of commission and omission which may have harmed the concrete after it passed from his control. Nevertheless, under circumstances where the concrete has actually failed to meet strength requirements (and where low tests are not merely indicative of improper sampling and testing), there may be no alternative but to accept core tests as the measure of the safety of the structure. The interpreter of the results of such tests should realize that there is no necessary relation between them and those for standard specimen. However, he should consider them as a valuable aid to engineering judgment.

Methods for obtaining and treating cores before testing are given in ASTM Methods of Obtaining and Testing Drilled Cores and Sawed Beams of Concrete (C 42). Meticulous observance of these procedures, in addition to considerable skill and caution beyond the requirements of the standard, is of the utmost importance in securing accurate tests.

Yield:

Tests for yield of concrete deserve special mention in connection with ready-mixed concrete. Ready-mixed concrete is purchased on a volume basis. Sometimes questions with respect to yield are raised because of the displacement of inadequately braced forms by the large batches. ASTM Test for Weight

Per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138) affords a satisfactory method for the determination of yield—one generally more accurate than measurement in the form.

Air Content:

Air-entrained concrete represents a large proportion of the volume of ready-mixed concrete. Control of the quantity of air is essential to the control of the consistency and strength of the concrete. Three different procedures for measuring air content are now on the books of ASTM—gravimetric, volumetric, and pressure.

The standard method of test for air content by the gravimetric method is described in ASTM Method C 138, previously referred to in connection with the test for yield. The gravimetric method has serious limitations, particularly as a field test, because of the necessity for precise knowledge of batch proportions, specific gravities, and so on. An error of 2 per cent in the moisture content of sand in a typical concrete mix, when the slump is not controlled, will cause the computed air content to be in error about 1 percentage while an error of 0.02 in specific gravity of aggregates will affect the computed air content by about one half of a percentage.

The volumetric procedure is described in the ASTM Method of Test for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173). This method is principally adapted to the laboratory, but it can be used successfully in the field. Briefly, it involves determination of the unit weight of concrete as in the gravimetric procedure but, instead of depending on computations, the absolute volume of ingredients other than air is measured directly by displacement in water.

The pressure meter method of test for air content is described in ASTM Test for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231). It is the procedure best adapted to ready-mixed concrete operations. The pressure method has a major advantage in that it does not require any knowledge of specific gravities or porportions of materials. The instrument should be calibrated for use at various localities if differences in altitude are considerable. It may yield erroneous results when relatively porous aggregates are used, since the application of pressure compresses not only the air within the mortar matrix but also within the pores of the aggregate. Some compensation for such errors may be made by applying a correction factor, but this procedure is not always satisfactorily accurate. The volumetric method is probably to be preferred for such determination.

CLOSURE

The ready-mixed concrete process has contributed efficiencies and economies which have led to greatly increased use of portland cement concrete. While the over-all quality of concrete has shown great improvement with time, the volume produced has increased so rapidly that the technology of production and handling has not always kept pace. Continued study should be made of the balance between speed and cost of production of the concrete and the control of its quality. Specifications for ready-mixed concrete, and concrete in general, need to be re-examined from the vantage of increased knowledge of manufacturing processes, of the significant factors which must be controlled, and of methods and procedures for effecting control—and this should be done with a thorough understanding of the respective responsibilities of the manufacturer and the purchaser.

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Special Categories

LIGHTWEIGHT CONCRETE AND AGGREGATES

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Both "lightweight concrete" and "lightweight aggregate" are general terms which include a wide variety of products and are frequently subject to varying definitions. Concrete lighter in weight than that usually obtained with "normal weight" aggregates (gravel, air-cooled blast-furnace slag, and crushed stone) may be produced by several methods:

1. Cellular or foam concrete, where the light weight is caused primarily by inclusion of a large amount of air or gas, usually 25 per cent or more. A suitable foam or foaming agent is combined with cement and water, either with or without other ingredients such as sand or pozzolanic fines.
2. No-fines or "popcorn" concrete, with the light weight produced by high air contents or specially graded aggregates having little or no fine material or both. Either lightweight or normal weight aggregates may be used.
3. Lightweight aggregate concrete, which is comparable to conventional concrete except for the use of lightweight materials as all or part of the aggregate.

Lightweight concretes produced by these various methods may range in unit weight from 15 or 20 lb/ft^3 to weights only slightly less than those of conventional concretes. Applications include:

(*a*) insulation or fill uses where light weight and a high degree of insulation are required, with strength being of lesser importance,

(*b*) masonry units requiring moderate strength with light weight and good insulation properties, and

(*c*) structural concrete for use in buildings and bridge decks, where weight reduction produces design economies and other characteristics desired are the same as for conventional concretes made with normal weight aggregates.

Lightweight aggregates of various types have been used for many years, but it is only recently that they have become an important factor in the concrete construction industry. Both mineral (inorganic) and vegetable (organic) materials of many varieties have been considered and tested for use as lightweight aggregates. The inorganic or mineral materials are the only ones that have been used to any significant extent and are the only materials covered by current ASTM specifications for lightweight aggregates. The most common method of making lightweight concrete in the United States is the use of lightweight mineral aggregates, with some use of cellular concretes for insulating and fill purposes.

Lightweight concrete, as discussed in this paper, is that having air-dry unit weights of 115 lb/ft^3 or less (plastic unit weights of about 120 lb/ft^3 or less) and made with lightweight aggregates as all or a major part of the aggregate. Discus-

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sion of lightweight aggregates is confined to the major types of inorganic aggregates covered by current ASTM specifications.

TYPES OF LIGHTWEIGHT AGGREGATES

Various methods have been used to classify lightweight concrete aggregates into general types. These are frequently based on original source, as either natural or artificial materials [*1*].[2] The artificial aggregates are either by-products of an industrial operation or specially processed by calcining, expanding, or sintering. In this classification system, such materials as pumice, scoria, and tuff are natural aggregates; by-product materials include cinders; processed lightweight aggregates are produced from clay, shale, slate, perlite, vermiculite, blast-furnace slag, and fly ash. Some of the materials may be placed in more than one of the type groups. For example, pumice may be used either in its original natural state or after heat processing (calcining); blast-furnace slag and fly ash are by-products that are processed for use as lightweight aggregates by expanding and sintering, respectively; clays, shales, and slates are natural materials that are always heat processed during production of lightweight aggregates.

It seems most reasonable to classify lightweight aggregate materials for discussion purposes on the basis of their end use under ASTM specifications. The various kinds of lightweight aggregates are, therefore, discussed here on the basis of the ASTM specifications in which they are included.

Lightweight Aggregates for Structural Concrete:

ASTM Specification for Lightweight Aggregates for Structural Concrete (C 330 – 64 T) covers "two general types" of lightweight, cellular, and granular inorganic material as follows:

"Aggregates prepared by expanding, calcining, or sintering products such as blast furnace slag, clay, diatomite, fly ash, shale or slate.

"Aggregates prepared by processing natural materials, such as pumice, scoria, or tuff."

Expanded shales, clays, slates and blast-furnace slags, and the natural scorias and pumices are the materials usually considered for structural lightweight concrete usage. Of these aggregates, the expanded shales, clays, and slates are the most readily available in many geographic areas and, therefore, are used in the majority of current structural applications. Slags and the natural aggregates are more limited in their areas of availability. Some use has been made of diatomite, while commercial production of fly ash as a lightweight aggregate is just beginning in the United States.

Expanded slag is produced by controlled processing and cooling of molten blast-furnace slag to obtain a lightweight cellular "clinker," which is subsequently crushed and screened to the required size. ASTM Definition of Terms Relating to Concrete and Concrete Aggregates (C 125) defines expanded blast-furnace slag as "The lightweight cellular material obtained by controlled processing of molten blast-furnace slag with water, or with water and other agents such as steam or compressed air or both." A number of different methods for accomplishing and controlling the expansion have been used [*2,3*], but no significant differences in product characteristics appear to result from them. Expanded slags have been produced in the United States since the late 1920's.

Expanded shales, clays, and slates are generally treated as a single type, since

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

no significant differences appear to exist due to the particular classification of the raw material used. Lightweight aggregates of this type were introduced in the United States about the time of World War I and have come into widespread use in recent years due to rising demands for lightweight concrete and widespread distribution of suitable raw materials. The aggregates are produced by agglomeration of suitable argillaceous material with or without bloating, being heated to the point of incipient fusion in a rotary kiln or on a traveling grate. After the material leaves the rotary kiln or traveling grate, it is cooled and then crushed and screened as necessary to obtain the desired sizes for shipment. Variations in the basic process also include the pre-forming of suitable materials to the desired sizes before introduction into the kiln so that the finished product does not need to be crushed but only screened.

Pumice and scoria are cellular, lightweight materials of volcanic origin. True pumice is a spongy lava from which steam or gas escaped while it was still molten. Commercially, volcanic ash naturally cemented together (more properly designated as tuff) is sometimes referred to as pumice. There are many deposits of pumice in the Western states, widely varying in strength and porosity characteristics. The weaker materials (including tuffs) are sometimes benefited by calcination at a temperature near incipient fusion. Scoria is a volcanic "cinder" with its pores chiefly in the form of vesicules instead of the more tubelike, interconnected pores of the pumices. It is available in a number of localities in the Western states, crushed and screened to proper sizes.

Lightweight Aggregates for Concrete Masonry Units:

ASTM Specification for Lightweight Aggregates for Concrete Masonry Units (C 331 – 64 T) includes all lightweight aggregates described above and, in addition, cinders and the expanded perlites and vermiculites. Since the primary use for perlite and vermiculite is in insulating concrete, they are covered in the following section on insulating concretes.

Cinders (referred to as "clinker" in Britain) are the residue of high temperature combustion of coal or coke. Known as "industrial cinders," "boiler cinders," or "steam cinders," these materials from industrial, power plant, and steam locomotive boilers were probably the first lightweight aggregates used in the United States. Christiansen [*4*] summarized their characteristics, uses, and performance in 1931, including possible effects from deleterious materials present in some sources. Dwindling supplies have resulted in greatly decreased usage, and applications in concrete masonry units constitute the only commercial use for cinders in concrete at the present time.

Lightweight Aggregates for Insulating Concrete:

ASTM Specification for Lightweight Aggregates for Insulating Concrete (C 332) covers those aggregates considered satisfactory for the manufacture of lightweight concrete not exposed to the weather and for which the primary consideration is the thermal insulating properties of the concrete. They are divided into two groups: Group I includes such materials as expanded perlite and vermiculite, which generally produce concrete weighing from 15 to 50 lb/ft^3 and having a thermal conductivity, k, of 0.45 to 1.50 Btu in./hr ft^2 deg F; while Group II includes the same materials as those covered under Specification C 330 for structural concrete (described above). When used in concrete primarily for insulation purposes, Group II aggregates are expected to result in concrete weighing from 45 to 90 lb/ft^3 and having thermal conductivities of 1.05 to 3.00 Btu in./hr ft^2 deg F.

Perlite is a volcanic glass which contains sufficient combined water that when it is heated quickly steam is generated internally and causes disruptive expansion and breakage into small expanded particles. Under controlled kiln conditions an aggregate of high internal void content and very low unit weight is produced.

Vermiculite is a micaceous mineral that forms another extremely lightweight aggregate when subjected to sudden heating. Conversion to steam of layers of combined water in the laminated structure of the mineral produces an "exfoliated" or expanded lightweight aggregate.

LIGHTWEIGHT AGGREGATE SPECIFICATIONS AND TESTS

The characteristics of lightweight aggregates covered by the current ASTM specifications are shown in Table 1 and discussed in the following sections. Specifications C 330 and C 331 are quite similar in requirements. Both include limitations on deleterious substances and various concrete making properties. Insulating concrete (Specification C 332) is presumed to be protected from weathering, and durability requirements are omitted; however, thermal conductivity is specified. Specific requirements are discussed below under the individual characteristics.

TABLE 1—LIGHTWEIGHT AGGREGATE CHARACTERISTICS COVERED BY ASTM SPECIFICATIONS.

Characteristics	ASTM Specification		
	C 330 (structural concrete)	C 331 (masonry units)	C 332 (insulating concrete)
Composition	S	S	S
Grading	S	S	S
Uniformity of grading	S	S	S
Unit weight	S	S	S
Uniformity of unit weight	S	S	S
Deleterious substances:			
Organic impurities	S	S	NS
Staining materials	S	S	NS
Clay lumps	S	S	NS
Loss on ignition	S	S	NS
Concrete making properties:			
Compressive strength and unit weight	S	NS	NS
Drying shrinkage	S	S	NS
Popouts	S	S	NS
Durability	S	S	NS
Unit weight and thermal conductivity	NS	NS	S

NOTE—S = specified; NS = not specified.

Composition:

All specifications stipulate that "the aggregates shall be composed predominantly of lightweight cellular and granular inorganic material," thus excluding from consideration such materials as seaweed, straw, cork, and sawdust, which have not been extensively used in this country [5]. General types of materials considered to be acceptable are enumerated and the aggregates described above are listed as examples. The specifications are not restricted to these particular aggregates, however.

Grading:

Each specification tabulates requirements for gradation of the aggregates. Coarse aggregate gradings up to 1 in. nominal maximum size are given in Specification C 330, while Specifications C 331 and C 332 restrict top size to $\frac{1}{2}$ in. for the same aggregates. Most lightweight aggregates are not available in sizes larger than $\frac{1}{2}$ or $\frac{3}{4}$ in., while perlites and vermiculites are normally supplied only in minus No. 4 gradations. Special gradings are stipulated in Specification C 332 for these two aggregates. In general, the gradings in Specifications C 330 and C 331 are similar to those for normal weight aggregates (ASTM Specification for Concrete Aggregates (C 33)) except for somewhat larger amounts of material passing the smaller sieve sizes. Specification C 331 provides that the gradation requirements may be waived to produce special characteristics in masonry units, such as texture, strength, weight, acoustical, or thermal insulating properties.

Grading is determined in accordance with ASTM Method of Test for Sieve or Screen Analysis of Fine and Coarse Aggregates (C 136), except that the sample size of fine aggregate is reduced (dependent upon unit weight), and the sieving time is limited to 5 min. Size of the test sample for coarse aggregate is set at 0.1 ft^3 min. These modifications are intended to prevent blinding the smaller sieves with an excessive volume of material and breakage of the more friable aggregates during the sieving operation.

Uniformity of Grading:

Variations in grading of lightweight aggregates may not only affect water demand and strength characteristics of concrete but will also change the unit weight because of differing specific gravities for various sizes of particles. The ASTM specifications for lightweight aggregates provide that the maximum variation in fineness modulus shall not be greater than 7 per cent from that of the sample submitted for acceptance tests, unless it can be demonstrated that the resultant concrete will have the required characteristics. This provision applies to both the coarse and fine aggregates.

Unit Weight:

All lightweight aggregates are subject to unit weight limitations as shown in Table 2. The tests are conducted in accordance with the Test for Unit Weight of Aggregate (C 29) using the shoveling procedure, except that oven-dry aggregate is tested. Minimum unit weights are stipulated for perlite and vermiculite to avoid excessively expanded, weak, and friable materials. Maximum values only are applied to the materials used in structural concrete and masonry units, since excessively light and weak aggregates of these types would be excluded from use either by failure to comply with other specification requirements or by the need for excessively high cement contents and the high concrete unit weights resulting from degradation.

TABLE 2—UNIT WEIGHT REQUIREMENTS FOR LIGHTWEIGHT AGGREGATES.

Type and Size	Dry Loose Weight, lb/ft^3 Min	Dry Loose Weight, lb/ft^3 Max
Expanded slags, clays, shales and slates, cinders and natural lightweights:		
Fine aggregate	...	70
Coarse aggregate	...	55
Combined fine and coarse	...	65
Perlite	7½	12
Vermiculite	6	10

The unit weight test furnishes a control over the maximum unit weight or density of lightweight aggregates and, therefore, provides some idea of the weight characteristics of concrete in which they might be used. However, correlations of aggregate and concrete unit weights are very general in nature, since particle shape and gradation differences may cause large variations in the voids between aggregate particles in the test.

Uniformity of Unit Weight:

Unit weight of a lightweight aggregate will be affected by changes in gradation, specific gravity or density of the mineral material, and in the particle shape or surface texture. The variations, of course, will be reflected in the water requirements, yield, unit weight, and strength of concrete in which the aggregate is used. To provide control of uniformity in use, the specifications require that the unit weight of successive shipments shall not differ by more than 10 per cent from that of the sample submitted for acceptance tests.

Deleterious Substances:

A number of materials are considered to be potentially deleterious in lightweight aggregates used in structural concrete or masonry units. Accordingly, they are subject to limitations in Specifications C 330 and C 331 but are not included in the insulating concrete aggregate specifications (C 332).

Organic impurities are tested for by ASTM Test for Organic Impurities in Sands for Concrete (C 40). Aggregates producing a color darker than the standard are subject to rejection, "unless it can be demonstrated that the discoloration is due to small quantities of materials not harmful to concrete."

Staining of concrete surfaces may result from use of lightweight aggregates that contain excessive amounts of iron or iron compounds. Although it is not a serious problem with the commonly used materials, the specifications provide a test procedure to evaluate the potential degree of staining from the aggregate.

The test consists essentially of exposing aggregate samples, enclosed in filter paper cups, to steam for 16 hr. The degree of staining is evaluated either by visual comparison of the stains on the filter paper with photographic reference standards provided in the specifications or by chemical determination of the amount of ferric oxide deposited on the filter papers from 200 g of the aggregate tested. Aggregates having a visual classification of "heavy stain" or darker are required to be tested by the chemical procedure and rejected if the Fe_2O_3 is 1.5 mg or more. The test method and specification limits are based on extensive studies of cinder aggregates by Seaton [*6*].

Clay lumps are limited to 2 per cent by weight of the aggregate in both Specifications C 330 and C 331. The test method used is ASTM Test for Clay Lumps in Natural Aggregates (C 142 – 64 T). Originally intended as a limit on the amount of "unburned or underburned lumps in expanded clay, shale or slate aggregates," it seems very doubtful that such lumps are sufficiently friable to be broken by finger pressure as stipulated in the test method. The specification wording was, therefore, changed from "unburned or underburned" to "clay" lumps, and the restriction is probably useful only for detecting contamination of the aggregate. Adequate evaluation of the effects of unburned or underburned lumps and development of a suitable test procedure for identifying them remains an unsolved problem. ASTM Recommended Practice for Petrographic Examination of Aggregates for Concrete (C 295) is useful in this regard.

Loss on ignition of lightweight aggregates is limited to 5 per cent for all materials covered by Specification C 330 and C 331, except cinders which are limited to 35 per cent (Specification C 331). No specific test procedure is required, nor is any reference made to such tests. Attention is called to the fact, in notes, that some lightweight aggregates may be partially hydrated during production or naturally contain innocuous carbonates or water of crystallization that would contribute to the loss on ignition. Such materials are not considered to be deleterious, and the notes state that "consideration should be given to the type of material when evaluating the product in terms of ignition oss."

Concrete-Making Properties:

Properties of the lightweight aggregates, other than those described previously, are specified indirectly in terms of their performance in concrete. These specification requirements are varied, dependent upon the primary use and desirable characteristics of the concrete. Requirements for drying shrinkage, popouts, and durability are listed in both Specifications C 330 and C 331, since both structural concrete and masonry unit performance could be adversely affected by lack of desirable properties in these respects. In Specification C 330, the importance of both strength and lightness of weight are recognized by suitable limitations. For insulating concrete, light weight and low thermal conductivity are of primary importance; limits are imposed on these characteristics in Specification C 332.

Drying shrinkage is limited to 0.10 per cent in Specifications C 330 and C 331 when determined in accordance with procedures outlined in the specifications. ASTM Test for Length Change of Cement Mortar and Concrete (C 157 – 64 T) is followed except that:

1. The concrete mix uses fixed proportions of one part cement to six parts aggregate by dry loose volumes, with sufficient water to produce a slump of 2 to 3 in.
2. Specimens are moist cured only 7 days with initial length measurements at the end of this period. Subsequent storage is at room temperature and 50 per cent relative humidity.
3. Shrinkage measurements are made at the ages of 28 and 100 days.

Although data proving these procedures to be the best test and specification limits seem to be lacking, no difficulties with excessive shrinkage in concretes made with aggregates known to pass the test requirements have been reported. Nevertheless, the procedure has an obvious drawback as an acceptance test for aggregates: the length of time required is 100 days after fabrication of the concrete test specimens. An accelerated test procedure for determination of the shrinkage is needed.

Popouts in lightweight concrete are comparatively rare but may occasionally be caused by such materials as unhydrated pieces of hard burned lime or magnesia, calcium sulfate, or unstable iron compounds. The popouts, when encountered, occur as a result of hydration or oxidation or both, rather than as a result of freezing and thawing action which is responsible for most popouts with natural aggregates. To test for such materials, concrete specimens are prepared in the same manner as outlined above for the shrinkage test. The specimens are then cured and tested in accordance with ASTM Test for Autoclave Expansion of Portland Cement (C 151). No popouts, as determined by visual inspection of the autoclaved specimens, are permitted under either Specifications C 330 or C 331.

Durability of either structural concrete or masonry units is important for those structures and structural elements exposed to weathering. To date, no specific procedures or specification limits have been developed to measure this attribute. Both Specifications C 330 and C 331 stipulate that "in the absence of a proven record of satisfactory durability" the lightweight aggregates "may be required to pass a concrete freezing and thawing test satisfactory to the purchaser." Details of the test procedures and the evaluation of results are left to the discretion of the purchaser.

Compressive strength and unit weight requirements included in Specification C 330 are shown in Table 3. The aggregate must be capable of producing concrete meeting one or more of the strength requirements, without exceeding the corresponding unit weights.

TABLE 3—STRENGTH AND UNIT WEIGHT REQUIREMENTS FOR STRUCTURAL CONCRETE MADE WITH LIGHTWEIGHT AGGREGATES.

Average 28-Day Compressive Strength, min, psi	Average Unit Weight, max, lb/ft³
4000	115
3000	110
2000	105

The specification provides that compressive strength be determined and specimens made in accordance with ASTM Test for Compressive Strength of Molded Concrete Cylinders (C 39) and Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192) except that moist curing is for only 7 days, followed by 21 days exposure to an atmosphere of 50 per cent relative humidity. Unit weight is determined from weights in air and immersed in water of cylinders made and cured in the same manner as those tested for compressive strength.

The unit weight thus obtained is an air-dried value that more closely corresponds to the weight in actual service than would the plastic unit weights. The change in curing procedure from the standard apparently has no significant effect on the 28 day compressive strength, as shown by investigations such as that of Shideler [*7*].

The specification, as originally adopted, included limitations on the absorption of the lightweight concretes, with the permissible values varying with the unit weight and strength values. Later investigations [*8*] demonstrated that absorption did not correlate with resistance to freezing and thawing and the requirements, therefore, were deleted.

Thermal conductivity values specified in Specification C 330 for insulating concretes are limited to 1.50 Btu in./hr ft^2 deg F for concrete having an oven-dry unit weight of 50 lb/ft^3 or less and to 3.00 for those weighing up to 90 lb/ft^3. The lighter concretes are those made with perlites and vermiculites; the 50 to 90 lb/ft^3 weights are those usually resulting from use of expanded shales, slags, and natural lightweight aggregates.

Thermal conductivity values are determined in accordance with ASTM Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (C 177). This test requires oven-dried specimens which are also used for unit weight determinations. Curing is for 7 days in the moist room followed by 21 days at 50 per cent relative humidity with subsequent oven drying. Unit weight is determined by weighing and measuring the dimensions of specimens having a volume of not less than 144 in.^{3}

Moisture content of insulating materials affects both the thermal conductivity and unit weight, and the use of oven-dried specimens does not duplicate conditions of actual use. The test conditions serve principally to permit general classification and comparison of materials and to provide a standardized reference condition. Under conditions prevailing in actual use, some increases in both weight and thermal conductivity should be expected from residual moisture in the job concretes.

Miscellaneous Tests and Properties:

In addition to the tests and properties included in the lightweight aggregate specifications and described previously, several others are of interest either because they are utilized in mix design and control or in specifications for normal weight aggregates.

Strength of the aggregate particles undoubtedly influences the properties of concrete made with them but is not usually included in specifications. The lightweight materials used for structural concrete and masonry units have demonstrated through experience that the degradation or breakdown during mixing and placing is not excessive and need not be a matter of concern with aggregates meeting the specification requirements for other properties. Attempts to correlate abrasion or crushing strength tests [*5*] of the aggregates with properties of the resulting concretes have been successful only in a very general manner. For a given type of lightweight aggregate (produced from similar raw materials) the strength of the aggregate varies roughly with the unit weight. The weaker lightweight aggregates are not, in general, capable of making the strength requirements for concrete included in Specification C 330. Perlites and vermiculites are much weaker structurally than the expanded or sintered aggregates but are employed only in the very light insulating concretes where high strength is not a requirement.

Although the strength of lightweight aggregates covers a wide range and undoubtedly overlaps the strength range for natural aggregates, the average is probably somewhat less than that for natural aggregates. Therefore, breakdown and degradation in handling is likely to be somewhat greater for the lightweights.

Chemical reactions of lightweight aggregates have not been a problem, and the specifications do not require that tests for chemical reactivity be applied to them. Mielenz [*9*] reports potentialities for alkali-aggregate reaction with some natural lightweight aggregates and expanded perlites but notes that the "volume change will not necessarily cause structural distress if appropriately accommodated in the design." The inherent characteristics of the aggregates and of the concretes made with them may also serve to reduce effects of such reaction—high porosity, low modulus of elasticity, and high creep in low strength insulating concretes might prevent structural damage in situations where normal weight concretes would be in distress. The fines in expanded shales, clays, and slates may react as natural pozzolans and could act to inhibit alkali aggregate reactions. Glassy phases of blast furnace slag are not deleteriously reactive [*9*] and in various investigations [*10,11*] have been shown to be effective inhibitors of such reactions.

Absorption and specific gravity test results for lightweight aggregates are quite different from those for normal weight aggregates. Bulk specific gravities of lightweight aggregates are much lower than those of normal weight aggregates, while absorptions are higher. Ranges of both bulk specific gravity and absorption for various types of lightweight aggregates have been summarized by Washa [*5*]. However, the highly irregular and vesicular surfaces of most lightweight aggregates makes surface drying difficult and uncertain, with resulting questionable validity of the absorption and specific gravity values. In addition to the methods provided in ASTM Tests for Specific Gravity and Absorption of Coarse and Fine Aggregates (C 127) and

(C 128), special techniques have been used by the Bureau of Reclamation [*5*], Saxer [*12*] and the Portland Cement Assn. [*7*] for determining the absorption and specific gravity of lightweight aggregates. Other test methods have been proposed by Helms and Bowman [*13*] and Landgren [*14*]. No method is widely used, however, and such tests are not usually conducted on lightweight aggregates.

While the natural aggregates do not usually exhibit any significant changes in specific gravity with varying size of particle, it has been shown [*15*] that specific gravity of lightweight aggregates increases with decreasing particle size. Changes in gradation, therefore, would be expected to change the specific gravity and absorption values. Higher specific gravities for the small sizes may also increase tendencies toward segregation during transportation and handling. Keeping the aggregate damp and handling in separated sizes are often recommended to minimize these tendencies.

Surface moisture tests, such as ASTM Test for Surface Moisture in Fine Aggregate (C 70), are of little value with lightweight aggregates, since accurate information on bulk saturated-surface-dry specific gravity is seldom available. Instead, the total moisture content is used in mix control and can be determined by procedures outlined in ASTM Test for Total Moisture Content of Aggregates by Drying (C 566 – 65 T).

PLASTIC LIGHTWEIGHT CONCRETE

Mix Proportioning:

The principles of mix proportioning usually applied to normal weight concretes are generally difficult to use with lightweight aggregates. The lack of accurate values for absorption, specific gravity, and free moisture means that net water-cement ratios cannot be established with sufficient reliability for use as a basis of mix proportioning. Variations in specific gravity with size of particle lead to the need for higher percentages of fine material than are usually considered desirable for normal weight concretes. Optimum air contents are usually somewhat higher than for normal weight concretes to avoid workability problems from the angular, vesicular-surfaced particles, to provide improved resistance to weathering, or to decrease unit weights in insulating and fill concrete uses.

Lightweight concrete mix designs are usually established by trial mixes, proportioned on the basis of cement content at the required consistency. ACI Standard 613A-59, Recommended Practice for Selecting Proportions for Structural Lightweight Concrete describes such procedures in detail. The proportions of fine and coarse aggregates and the cement and water requirements are estimated or based on previous experience with the particular aggregate. The dry loose unit weights and moisture contents of the aggregates are determined and the first mix made on the basis of the estimated proportions and sufficient water to obtain the desired slump. Unit weight and air content of the mix are determined. Since weights of all materials in the mix and the specific gravities of all except the lightweight aggregates are known, a "specific gravity factor" for the aggregate can be calculated. This is not a true specific gravity value, since the proportions of free and absorbed water are unknown, but it provides a useful value for adjusting mix proportions for additional trial batches. A pycnometer method for determining the specific gravity factor has recently been developed and reported by Landgren, Hanson, and Pfeiffer [*16*]. Final mix design is on the basis of dry, loose volume of aggregate per cubic yard, total water necessary to provide the desired slump, and the cement content necessary to obtain the required strength.

Mixing Procedures and Control:

Mixing procedures for lightweight concretes may vary with the different types of aggregates, and the producers' recommendations should be followed. However, the general practice for structural and masonry unit concretes is to mix the aggregates and about two thirds of the mixing water required for periods up to one minute prior to the addition of the cement, air-entraining agent, and the balance of the mixing water. Mixing is then continued as required for homogeneity, usually two or more minutes for structural mixes and four or more minutes for masonry unit mixes. For some insulating concretes, the aggregate is added last to minimize degradation.

Control of lightweight mixes is obtained by keeping the cement content, slump, and volume of dry aggregate per cubic yard constant. Unit weight, slump, and air content tests of the fresh concrete will determine if changes in aggregate weight have occurred and if the proper cement content is being used. If a change in aggregate weight has occurred, checks of moisture content, gradation, and unit weight of the aggregate can be made to determine causes of the change.

The slump should not only be maintained as consistent as possible but also kept as low as is practical for the placing conditions. Over-wet lightweight mixes tend to segregate, and the lightness of the larger particles causes them to float to the surface where they may interfere with finishing operations. Overmanipulation, excessive vibration, and so on should be avoided to minimize such tendencies.

Plastic Concrete Tests:

Tests of plastic or freshly mixed concretes conducted by standard procedures are confined principally to the structural lightweight concrete mixes.

Workability is determined by means of ASTM Test for Slump of Portland Cement Concrete (C 143). The test results are not directly comparable to those obtained with normal weight aggregates, that is, a 3-in. slump does not indicate the same workability or placeability for a lightweight concrete as it would for a normal weight mix. For the same workability, a lightweight mix would have a lower slump, due to the lesser weight of concrete causing distortion of the mass upon removal of the slump cone.

Unit weight of freshly mixed structural lightweight concrete is important in determining yield and controlling mix proportions. ASTM Test for Weight per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138) is used. With lightweight concretes, tapping the sides of the measure to remove air bubbles is particularly important, since holes left by rodding do not fill as readily as in the case of normal weight mixes. The calculations shown in the method for gravimetric air content are not applicable to lightweight mixes, because the total absolute volume of the mix ingredients cannot be accurately determined.

Air content of lightweight concretes is determined by ASTM Test for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173). This method is applicable to any type of aggregate, whereas pressure methods are accurate only for denser aggregates where proper correction for air within the aggregate particles can be readily made. Porosity characteristics and difficulties in specific gravity determinations of lightweight aggregate make both the pressure and gravimetric methods very inaccurate when used on lightweight concretes. ASTM C 173 volumetric method gives the most reliable results for air content of lightweight concretes and is, therefore, the recommended procedure.

HARDENED LIGHTWEIGHT CONCRETE

Earlier published information on the characteristics and properties of lightweight concretes (and lightweight aggregates) was summarized by Washa [*5*], Kluge [*17*], Carlson [*18*], and Valore [*19*], the last three of these covering lightweight structural concrete, masonry units, and insulating concretes, respectively. In many cases, tests for properties of lightweight concretes are the same as those for concretes of normal weight, while in others special tests particularly suited to lightweight concrete characteristics are used.

Unit Weight:

Except in special design circumstances, the unit weight of hardened normal weight concrete is seldom of interest. Lightweight concretes, however, are often economical in use principally because of the weight savings involved. Specifications for lightweight structural concrete usually stipulate a maximum unit weight in an air-dry condition, presumed to be comparable to weight in service. ASTM Test for Unit Weight of Structural Lightweight Concrete (C 567 – 65 T), provides a test procedure for determining this value. The usual range of air-dry unit weight values for structural lightweight concrete is from 85 to 115 lb/ft^3.

Unit weights of insulating concretes are usually determined in an oven-dry condition, using the oven-dry weight and dimensions of companion specimens to those subjected to either thermal conductivity or compressive strength tests. Insulating concretes made with perlite or vermiculite aggregates or cellular concretes may range from 15 to 50 lb/ft^3, while those made with other types of lightweight aggregate are usually in the 50 to 90 lb/ft^3 range.

Unit weight of concrete in masonry units is on an oven-dry basis for all aggregates. The method of test in Methods of Sampling and Testing Concrete Masonry Units (C 140 – 63 T) is used, with lightweight units having concrete weights usually in the range of 75 to 110 lb/ft^3.

Strength Properties:

The strength of lightweight concretes varies with unit weight, aggregate characteristics, cement content, and all other factors that affect strength of any concrete. It is quite low for insulating and fill concretes while structural concretes compare quite favorably with excellent quality conventional concretes.

Compressive strength of both lightweight structural and masonry unit concretes are determined by the same procedures used for testing normal weight concretes. Although tests in an air-dry condition are stipulated in Specification C 330 for evaluation of concrete making properties of the lightweight aggregates, these procedures are not intended for use in job control or acceptance testing of lightweight concretes. Standard procedures for making, curing, and testing normal weight concretes are used for these purposes. Specifications usually require the lightweight concretes to have the same compressive strengths as would be required for normal weight concretes in structural and masonry unit uses. Although higher cement contents are required with some lightweight aggregates to obtain these equivalent strengths, many of them have essentially the same cement factor-strength relationships as do high-quality normal weight aggregates.

In contrast, insulating concretes are not required to produce high compressive strengths and frequently are in the range of 200 to 800 psi. ASTM Test for Compressive Strength of Lightweight Insulating Concrete (C 495 – 62 T) covers the procedures for testing fresh concretes weighing less than 50 lb/ft^3. Either 4-in. cubes or 3 by 6-in. cylinders are used, with curing consisting of 7 days moist, 18 days at 50 per cent relative humidity and 3 days in an oven at 140 F. Moisture content has a large effect on the strength of insulating concrete, and complete saturation would not represent its "in service" condition. The oven-dry test requirement was selected as a reproducible condition that would provide reasonably realistic values. Tendencies of some insulating concretes to crack if dried rapidly led to the use of relatively low-temperature oven drying preceded by a period of air drying. A similar test condition is stipulated for determining compressive strength of hardened insulating concrete, using cubes cut from structures in place (ASTM Method for Securing, Preparing, and Testing Specimens from Hardened Lightweight Insulating Concrete for Compressive Strength (C 513 – 63 T)).

Flexural strength tests are sometimes conducted on structural lightweight concretes, using the standard test procedures employed for normal weight materials. The ratio of flexural strength to compressive strength is usually somewhat higher for the lightweight concretes than for regular concrete mixes [*26*].

Tensile strength of lightweight structural concrete is determined by ASTM Test for Splitting Tensile Strength of Molded Concrete Cylinders (C 496 – 64 T). For lightweight concretes, the method provides in Section 3(b) that the test shall be run on air-dried cylinders for evaluation in accordance with ACI Standard 319-63, Building Code Requirements for Reinforced Concrete. This deviation from standard moist curing is based on investigations [*21*] that show better correlation of diagonal tension strengths with air-dry tensile strengths than with tests of moist cured specimens.

Plastic Properties and Creep:

These tests on lightweight concretes are usually confined to the structural concretes. The standard test procedures, ASTM Test for Static Young's Modulus of Elasticity and Poisson's Ratio in Compression of Cylindrical Concrete Specimens (C 469) and ASTM Test for Creep of Concrete in Compression (C 512 – 65 T), are used. The modulus of elasticity is usually lower than that of normal weight concrete of the same strength, rarely exceeding 2,500,000 psi. The value varies with both unit weight and strength. Pauw [*22*] developed a relationship between modulus of elasticity, unit weight, and strength that appears applicable to both lightweight and normal weight concretes. The current ACI Building Code 318-63 uses this relationship to obtain design values for lightweight structural concretes in the absence of actual test values. The influence of unit weight is apparently the reason for the lower E values of lightweight concretes.

Some investigations [*7*] have shown lightweight structural concretes to have higher creep values than normal weight mixes. Such comparisons, however, have usually been based on comparative tests using only one normal weight aggregate, frequently of higher than average quality. Although the average lightweight concrete may have greater creep than the average normal weight concrete, the variations found in both types results in considerable overlap in the ranges of values.

Volume Change:

The volume change of lightweight concretes is usually somewhat greater than that of normal weight concrete having similar cement contents. However, some of the structural lightweight concretes have drying shrinkage characteristics that fall in the range found in concretes made with regular aggregates.

High drying shrinkage alone is not indicative of increased cracking tendencies, since resistance to cracking is also dependent upon modulus of elasticity, tensile strength, and creep characteristics. Lightweight concretes are generally capable of undergoing greater deformation before cracking than regular concrete. Shrinkage tests may be conducted in accordance with Method C 157 although, as noted previously, this test has been modified in some details for use in determining the concrete making properties of lightweight aggregates (Specification C 330).

Volume changes due to temperature variations are quite similar to those of regular concretes. The coefficient of thermal expansion is about the same for lightweight and normal weight concretes, except in the case of lightweight masonry units where the value is usually smaller.

Thermal Conductivity:

The thermal conductivity of lightweight concrete varies primarily with unit weight; however, as shown by Foxhall [*23*], type of aggregate is also a significant factor. Tests are usually conducted by Method C 177, as discussed previously under the specifications for lightweight aggregates for insulating concrete. Values of thermal conductivity of lightweight concretes indicate that structural concretes have about one third, masonry units about one fifth, and insulating concretes about one tenth of the thermal conductivities of regular concretes.

Fire Resistance:

Lightweight concretes offer greater fire resistance than do normal weight concretes in structural uses, fireproofing, and masonry units [*24,25*]. Comparison of the results of standard fire tests on lightweight and normal weight concretes indicates that 2 in. of lightweight concrete is the equivalent of about a 3 in. thickness of normal weight concretes. In both cases, of course, the type of aggregate has a significant effect upon the fire resistance of the concrete.

Bond with Reinforcing Steel:

Bond characteristics of lightweight structural concretes may be evaluated by means of ASTM Test for Comparing Concretes on the Basis of the Bond Developed with Reinforcing Steel (C 234). Tests reported by Shideler [*7*] show bond strengths of lightweight concrete to be essentially the same as those of normal weight concrete.

Corrosion of Reinforcing Steel:

Both natural and manufactured lightweight aggregates are free from any corrosive effects on reinforcing steel. Cinder concrete used in the past was questioned in this respect, but excellent records in many places led Christensen [*4*] to the conclusion that corrosion of the reinforcing steel was associated principally with the use of porous and permeable, low-quality concrete exposed to moist conditions. Lightweight aggregates of the types now commonly employed in structural concrete have been in use many years without any evidence of ill effects on the reinforcing steel. Some lightweight concrete exposed to sea water for long periods and with less than an inch of concrete over the steel [*26*] has shown no evidence of corrosion. Short [*27*] concluded, as a result of corrosion tests and experience, that aggregate type (dense or lightweight) had little, if any, influence on rusting of the reinforcement, with thickness of cover, quality of the concrete, and exposure conditions constituting the important factors.

Absorption:

The water absorption of lightweight concretes is greater than that of concretes made with normal weight aggregates. For valid comparison of absorption values they must be expressed as percentages by volume rather than by weight, due to the wide differences in unit weights of the concretes. No correlation of absorption with any significant or desirable property of the concretes has been shown to exist, and no limits are placed on light weight structural or insulating concretes. Specifications for concrete masonry units, however, do include such limitations where exposure to weathering is involved. The maximum permissible absorptions, on a volume basis, are varied with both severity of exposure and with the unit weight of concrete in current specifications. Masonry units made with the lighter weight, generally more absorptive aggregates, are permitted higher absorption values than heavier units containing denser aggregates.

Acoustic Properties:

Sound absorption properties of building materials are of interest in control of air-borne noise in many structures. Porosity is an important factor in the sound absorbing properties, and concrete masonry units are frequently employed in such uses. Lightweight aggregates apparently contribute to sound absorbing efficiency in masonry units [*18*], generally showing higher efficiency than normal weight concrete units. However, continuous pores through such units tend to provide lower resistance to sound transmission through walls. There would appear to be little difference between sound transmission through walls of lightweight structural concrete, or of lightweight masonry units sealed by painting or plastering, and walls of normal weight concrete.

Nailability and Sawability:

Lightweight concretes are more readily nailable and can be sawed more easily than normal weight concretes, particularly compared to those made with very hard, natural aggregates. In general, the lower the unit weight, the better the nailability and sawability. Thus insulating concretes are very easily sawed with the lightweight structural concretes being somewhat more difficult to saw or nail.

Durability:

Although laboratory studies have shown that some lightweight structural concretes with inadequate entrained air [*8*] and lightweight masonry units made with lean mixes [*28*] can be destroyed by artificial cycles of freezing and thawing, properly proportioned mixes are indicated to have excellent durability. The field performance of all types of lightweight concrete appears to have been generally excellent from the standpoint of resistance to the effects of weathering. The literature seems devoid of any authenticated field cases where lack of durability has been caused by use of lightweight aggregate concrete.

It seems that the possible use of lightweight aggregates containing excessive amounts of unburned or underburned particles of clay or shale is the greatest potential danger to durability in lightweight structural concretes. Such particles could retain sufficient clay-like characteristics to cause high drying shrinkage and detrimental volume changes from alternate wetting and drying in structural concrete. The drying shrinkage test included in the lightweight aggregate specifications, ASTM C 330, should preclude the use of such aggregates. However, this phase of lightweight concrete durability is deserving of additional research and investigation.

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Special Categories

PACKAGED, DRY, COMBINED MATERIALS FOR MORTAR AND CONCRETE

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The packaging of dry, combined materials for concrete and mortar was initiated in this country about 1936. A trade name was copyrighted. A process for proportioning, mixing, and packaging was developed, but its details were not made public. A considerable part of the specialized equipment used was patented. Licenses to use the process, special equipment, and trade name were issued to a number of companies under an agreement providing for details of packaging and marking, quality control, and similar items.

The business grew slowly at first, but quite rapidly since the end of World War II, and quickly attracted the attention of others. Since there were many approaches to proportioning and mixing and since many packaging methods were available, many competitors of the original licensees and many new trade names quickly appeared. Current estimates of the number of producers now operating vary from less than 100 to about 200. Annual production is generally estimated to be more than twenty million packages.

The Products

As the name indicates, "Packaged, Dry, Combined Materials for Mortar and Concrete" are combinations of cementing materials and aggregates which require only mixing with proper quantities of water to be ready for use. The packages, in weights ranging from 10 to 90 lb, are generally available at the producing plant, building supply and hardware stores, as well as some supermarkets. The general trend is to thoroughly mix the various ingredients before packaging in moisture resistant multiwall paper bags. Some producers introduce the ingredients into the package separately without prior mixing. Still others package the cement separate from the aggregates. Due to the adoption of ASTM Specification for Packaged, Dry, Combined Materials for Mortar and Concrete (C 387) in 1956 and subsequent revisions of that specification covering these materials which will be discussed later in this paper, the buying public has become increasingly specification conscious. The disparity in companies and their products which characterized the industry during its early expansion is rapidly decreasing.

Use of the Products

The developer of the packaged materials for concrete and mortar visualized the principal user as the "do-it-yourself" homeowner. While this represents an important market, the scope of use has expanded far beyond it. Industry makes use of the products for repairs and

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maintenance requiring relatively small quantities of materials. Highway departments and railroads use them for patching, setting signs, signals, and so on. Public utilities are regular customers, using the concrete or mortar for a variety of purposes including patching of sidewalks and pavements after installation of pipe or conduit, anchoring poles carrying telephone cables and transmission lines, and for other work too numerous to list. In other cases, concrete in quantities of several cubic yards up to several hundred cubic yards has been used in locations inaccessible to ordinary transport.

QUALITY CONTROL

There has been no uniform approach to the control of quality of these packaged products for mortar and concrete. A most important step in the direction of standardization and quality control was taken when ASTM, urged by producers of the packaged materials, developed ASTM Specification C 387. This was first published as "proposed" in 1955, adopted as tentative in 1956, revised in 1958, adopted as standard in 1960, and further revised in 1964.

This specification now requires that cements, admixtures, and aggregates, and also test methods conform with ASTM standards. They cover: (1) two classes of lightweight concrete, (2) two classes of normal weight concrete, (3) a high-strength concrete-sand mortar, and (4) four classes of mortar for unit masonry. Minimum compressive strength is specified for each product. For masonry mortars, minimum water retention is also specified (see Table 1).

The specification also covers containers, qualities of materials for use in constructing the containers, requires that all packages be identified as conforming to ASTM Specification C 387 and that there be printed on the bag:

(*a*) kind and type of material used,

(*b*) net weight in each bag,

(*c*) yield in cubic feet or square feet per inch of thickness, and

(*d*) amount of water recommended for mixing.

TABLE 1—PHYSICAL REQUIREMENTS.

Kind of Material	Water Retention, min, %	Compressive Strength, min, psi			
		1 day	3 days	7 days	28 days
Mortar for unit masonry:					
Type M	70	...	...	...	2500
Type S	70	...	...	...	1800
Type N	70	...	...	...	750
Type O	70	...	...	...	350
Concrete mortar	...	...	...	3000	4000
Normal strength concrete	...	...	...	2500	3500
High early strength concrete	...	...	3000	4000	...
Lightweight concrete (using natural sand)	...	...	...	2000	3000
Lightweight concrete	...	...	...	1000	1500

SIGNIFICANCE OF C 387

In ASTM Specification C 387, the producer and consumer of packaged materials for mortar and concrete have a powerful tool for bringing about standardization and improvement of products. The frequent revisions of the original 1956 specification indicate the widespread interest in the subject. The principal purpose of such revisions has been to clarify statements in the 1965 specification, rather than to make any great changes in its original requirements. Further changes will undoubtedly be made in the future.

Both producer and consumer should scrutinize Specification C 387 to determine what changes or additions seem to be desirable. ASTM is so organized that recommendations from any source will be considered by the sponsoring committee. The responsible producer may be presumed to comply with the basic requirements of Specification C 387 and to use the procedures outlined to study his products and to develop higher and more uniform quality. The consumer should be encouraged to require that the product be certified to comply with Specification C 387. A product made with the letter and spirit of this specification will be a good product. There is now little need to accept anything less.

Acknowledgments:

In the absence of any known prior publications or comprehensive surveys of the industry discussed, the writer gratefully acknowledges the suggestions and assistance of several members of the sponsoring subcommittee, and especially the aid of Honorary Member Stanton Walker in formulating and presenting the contents of this paper.

PART III

Tests and Properties of Concrete Aggregates

STP169A-EB/Apr. 1966

Concrete Aggregates

PETROGRAPHIC EXAMINATION

By Richard C. Mielenz,[1] Personal Member, ASTM

Petrographic examination of concrete aggregate is visual examination and analysis in terms of both lithology and properties of the individual particles. The procedure requires use of a hand lens and petrographic and stereoscopic microscopes; less commonly, X-ray diffraction or differential thermal analysis is used to supplement the microscopical examination. By petrographic examination, the relative abundance of specific types of rocks and minerals is established; the physical and chemical attributes of each, such as particle shape, surface texture, pore characteristics, hardness, and potential alkali reactivity, are described; coatings are identified and described; and the presence of contaminating substances is determined.

As will be discussed subsequently, petrographic examination contributes in several ways to the investigation, selection, testing, and control of aggregates. Consequently, the method is being progressively more widely applied. Since 1936, all aggregates used in concrete construction by the Bureau of Reclamation have been examined petrographically as a part of the basis for their selection [*1*].[2] The method has been applied similarly by the Corps of Engineers since before 1940 [*2*]. Coarse aggregates proposed for use in either portland-cement concrete or bituminous concrete are examined by petrographic methods in laboratories of the Ontario Department of Highways, and specifications governing acceptance are based upon the results [*3*]. Petrographic examination of aggregates is performed also by several other agencies of the U.S. government and state highway departments, and may be obtained through some commercial laboratories.

In 1952, the ASTM accepted a Tentative Recommended Practice for Petrographic Examination of Aggregates for Concrete (C 295), which was adopted as a standard in 1954. Minor modifications were made in 1964. Petrographic examination is cited in the ASTM Specifications for Concrete Aggregates (C 33).

The abundant data obtained and the rapidity with which petrographic examination can be completed justify more general use of the method in investigation, selection, manufacture, and use of concrete aggregate.

This paper summarizes the objectives and applications of petrographic examination of aggregates with reference to gravel, sand, crushed stone, slag, and the most common types of lightweight aggregate. Techniques of the examination are treated very briefly because satisfactory instructions have been published

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

[*1–10*] and are included in ASTM Recommended Practice C 295.

PURPOSE OF THE PETROGRAPHIC EXAMINATION OF CONCRETE AGGREGATES

Preliminary Determination of Quality:

Preliminary petrographic examination of concrete aggregate is performed either in the field or in the laboratory as an adjunct to geologic examination, exploration, and sampling. The examination assists the geologist or materials engineer in determining the extent to which consideration of an undeveloped deposit is justified. Also, the preliminary petrographic examination indicates the relative quality of aggregates from alternate sites. By revealing variations in the material, examination of exposures or core from pilot drill holes establishes the minimum program of exploration and sampling necessary for acceptance or rejection of the deposit.

Establishing Properties and Probable Performance:

Petrographic examination is primarily a supplement to the acceptance tests. Probable performance of concrete aggregate is estimated in two general ways by petrographic examination. First, the examination reveals the composition and physical and chemical characteristics of the constituents. From this information, the probable response of the aggregate to such phenomena as attack by cement alkalies, freezing-thawing, wetting-drying, and heating-cooling, usually can be estimated. The rapidity with which the petrographic examination predicts potential alkali reactivity of aggregate is especially valuable because of the long time commonly required by tests of concrete or mortar.

Second, petrographic examination establishes the fundamental nature of aggregates so that aggregates from unfamiliar sources can be compared with aggregates upon which information is available. This application is discussed below.

Correlation of Samples with Aggregates Previously Tested or Used:

Detailed petrographic examination is the only test that permits comparison and correlation of samples with aggregates previously used or tested. Thus, data and experience previously obtained by use and long-time tests of similar aggregates can be applied in the selection of materials proposed for current work, even though the materials come from new sources.

Courtesy of the Michigan State Highway Dept.

FIG. 1—Popouts produced by claystone, shale, and chert in concrete pavement near Jackson, Mich.

By relating the sample to aggregates previously used in construction, aggregate indicated to be unsound by standard tests may be found adequate, or conversely, aggregate indicated to be sound in standard tests might be found unsatisfactory for the intended use.

For example, gravel in certain deposits near Jackson, Mich. meets usual specification requirements for soundness, abrasion resistance, and content of soft particles, yet produces objectionable popouts in pavements after exposure for two winters (Fig. 1). Petrographic examination of the gravel and of particles producing popouts during service has identified the unsound rock types. Examination of proposed materials will demonstrate the presence or absence of these rock types and thus will indicate, in the light of other data, whether the aggregate should be accepted, rejected, or subjected to special tests.

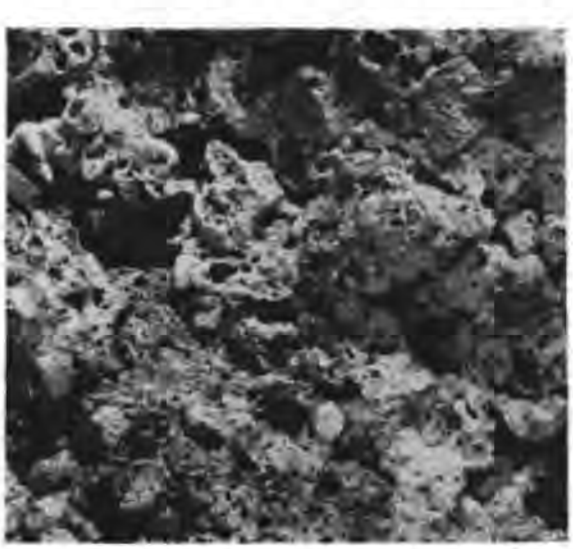
Selecting and Interpreting Other Tests:

All properties of aggregates influencing performance of concrete ordinarily are not evaluated by test before selection of the aggregate to be used in the work, primarily because of cost and time required. Such factors as thermal properties and volume change with wetting-drying rarely are determined. Other properties, such as chemical reactivity or effect of the aggregate on the freezing-thawing resistance of concrete, usually are not determined. Consequently, it is worthwhile to apply a test by which the relative significance of such properties can be determined and the need for supplementary quantitative tests indicated.

Petrographic examination aids interpretation of other tests. For example, are the particles identified as clay lumps in accordance with ASTM Test for Clay Lumps in Natural Aggregate (C 142 – 64 T) indeed clay lumps or are they merely friable or pulverulent particles? What is the cause of unexpected failure of concrete specimens in freezing and thawing? Is it the presence of unsound particles which do not disintegrate in the sulfate soundness test, yet expand in freezing and thawing? Is it the result of alkali-aggregate reaction? Is failure in the soundness or abrasion test the result of complete breakdown of a small proportion of unsound or soft particles or partial disintegration of the greater proportion of the aggregate?

Detection of Contamination:

Petrographic examination is the best method by which potentially deleterious and extraneous substances can be detected and determined quantitatively. Inadvertent contamination with natural substances, industrial products, or wastes, such as from overburden or from trucks or railroad cars not properly cleaned of previous cargo, may decrease the quality of aggregate markedly. Contamination introduced by containers may invalidate samples. Such substances as clay, soil, coal, vegetable matter, petroleum products, or refractories containing calcium or magnesium oxides are especially important. Incomplete processing of synthetic aggregates may contaminate the finished product with raw or partially fired raw materials or coal (Fig. 2).



Courtesy of the Bureau of Reclamation, U.S. Department of Interior

FIG. 2—Coal in sintered clay before crushing (×2.7). Reduced one half for reproduction.

Undesirable substances inherent in the material, such as coatings, clay, plant remains, coal, and soluble salts, are detected easily and can be determined quantitatively by petrographic methods.

Determining Effects of Processing:

Petrographic examination aids in production and processing of aggregate. The relative merit of alternative processing methods and equipment can be determined quickly by comparison of the original material with the processed aggregate. Comparison can be based on particle shape, content of unsound or chemically reactive constituents, presence of coatings, or production of rock dust.

The feasibility of beneficiation by removal of unsound or deleterious constituents depends on the properties of the particles and their abundance. By petrographic examination, the undesirable particles can be identified and separated. Their properties then can be evaluated and compared with properties of the remainder of the aggregate. If the undesirable particles are unusually soft, friable, dense, lightweight, or high in magnetic susceptibility, separation may be feasible on a commercial scale.

Petrographic examination can be used to control the manufacture of synthetic aggregate, such as expanded shale or clay, perlite, slag, and other types. Microscopical examination will reveal the presence of raw or underburned materials, alkali-reactive phases, and contaminants, such as coal. X-ray diffraction analysis is the most dependable means to identify and determine quantitatively the proportion of crystalline phases, such as periclase (MgO), free lime (CaO), and clays whose crystal structure was not destroyed by the calcination. Differential thermal analysis can be used to estimate the effective temperature to which clays, shales, slate, and similar materials have been fired; the trace of the differential thermal curves for the raw and fired product will coincide above the effective temperature achieved in the firing operation.

PERFORMANCE OF THE PETROGRAPHIC EXAMINATION

Samples for Petrographic Examination:

Samples of aggregate for petrographic examination should be representative of the source. Recommended procedures for sampling and for preparation of the sample for analysis are covered by ASTM Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for use as Highway Materials (D 75) and Recommended Practice for Petrographic Examination of Aggregates for Concrete (C 295).

Examination in the Field:

Petrographic analysis of samples in the field usually is qualitative or only semiquantitative because lack of facilities makes detailed work difficult. However, detailed examination in the field may be warranted if aggregate retained on the 3-in. sieve is to be used in the work, inasmuch as a representative sample of this size fraction necessarily weighs several hundred pounds and transportation of the sample to the laboratory is costly. Chips from cobbles not adequately identified in the field should be taken to the laboratory for further study.

If the deposit or rock exposure is variable, samples should be selected from each zone, and detailed notes made at the site should relate each sample to a particular zone and portion of the deposit or rock ledge. The relative proportion of unsound, fractured, or chemically deleterious materials should be estimated from measurements made at exposures. These notes and the results of the tests on the samples will be the basis for operation of the deposit inasmuch as it may be desirable to waste or avoid zones

FIG. 3—Performing the petrographic examination of fine aggregate with stereoscopic microscope, mechanical stage, and tally counter.

or portions containing inferior or unsuitable materials.

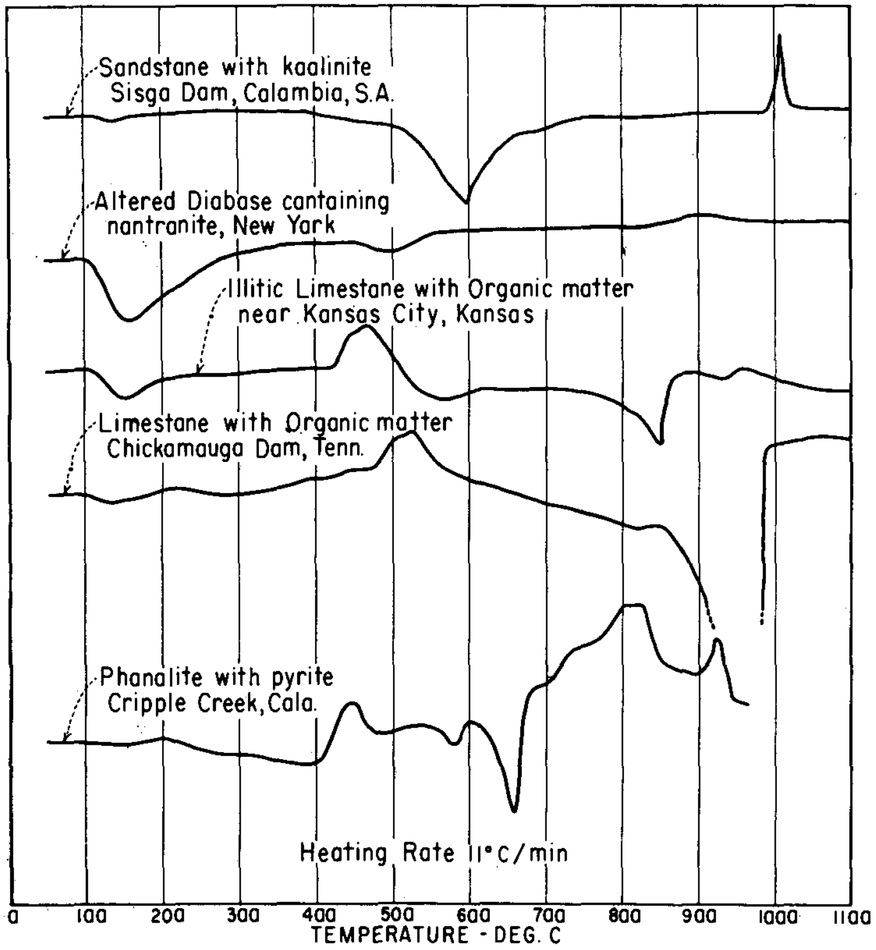
Examination in the Laboratory:

Petrographic examination of aggregate in the laboratory may be brief or detailed. Brief examination indicates the relative merit of materials from alternate sources and supplies justification for abandonment or continued investigation of undeveloped deposits. The preliminary examination should not replace the quantitative analysis included in the program of acceptance tests.

Samples supplied to the laboratory comprise: (1) granular materials, such as gravel, sand, crushed stone, slag, or synthetic aggregate; (2) stone in quarried blocks and irregular pieces; or (3) drilled core. For granular materials, the examination should be performed on all size fractions included in the aggregate. The sample of each size fraction should comprise a minimum of 300 particles. For natural sand and gravel and crushed stone, the minimum representative samples are:

Size Fraction	Weight of 300 Particles
3 to 1½ in.	57 lb
1½ to ¾ in.	19 lb
¾ to ⅜ in.	2.6 lb
⅜ to 3/16 in.	0.75 lb
No. 4 to 8	15 g
No. 8 to 16	2.1 g
No. 16 to 30	0.28 g
No. 30 to 50	0.033 g
No. 50 to 100	0.0066 g

Samples for petrographic examination are obtained by sieving in accordance with the ASTM Test for Sieve Analysis of Fine and Coarse Aggregates (C 136) and Tests for Amount of Material Finer Than No. 200 Sieve in Aggregates (C 117). The fractions are quartered or, for fine aggregate, split repeatedly on an appropriate riffle. Specially calibrated and fabricated cups holding 100 grains in the various size fractions have proved convenient in the Petrographic Laboratory of the Bureau of Reclamation for obtaining the small samples of fine aggregate for the analysis.



Courtesy of the Bureau of Reclamation, U.S. Department of Interior

Kaolinite is indicated by the endotherm (downward shift) at 990 to 1025 C. Nontronite is revealed by endotherms at 100 to 350 C and 450 to 550 C. Illite produces the small endotherms at 100 to 200 C and 500 to 615 C. Organic matter produces large exotherms at 430 to 500 C or 440 to 600 C. Pyrite develops a marked exotherm at 400 to 485 C.

FIG. 4—Typical differential thermal analysis records obtained on concrete aggregates.

Details of procedure are outlined by Mather and Mather [*2*] and in ASTM Recommended Practice C 295.

During the analysis, helpful clues to identity and physical condition can be obtained by noting such features as: (1) friability or pulverulence in the fingers; (2) resonance when struck; (3) ease of fracturing; (4) nature of the fracture surface and fracture fillings; (5) odor on fresh fracture; (6) color and its variation; (7) internal structure, such as porosity, granularity, or lamination; and (8) reaction to water, such as absorption of droplets on fresh fracture, evolution of air on immersion, capillary suction against the tongue, slaking, softening, or swelling.

Fractions retained on the No. 30 sieve are best identified, examined, and counted under the stereoscopic microscope. The analysis can be made conveniently by traversing a representative portion of each fraction by means of a mechanical stage and microscope assembly like that employed in the point-count procedure according to ASTM Recommended Practice for Microscopical Determination of Air-Void Content, Specific Surface, and Spacing Factor of the Air-Void System in Hardened Concrete (C 457 – 60 T) (Fig. 3). The mineralogic composition of finer fractions usually is determined most easily and accurately in immersion oils under the petrographic microscope. However, better continuity in description of physical characteristics of the particles is obtained if analysis of fractions passing the No. 30 sieve and retained on the No. 100 sieve is performed under the stereoscopic microscope.

Thin sections occasionally are necessary in examination of natural aggregate. They usually are employed in the study of quarried stone. Thin sections or polished surfaces always should be used in analysis of blast-furnace slag. X-ray diffraction and differential thermal analysis may be required to identify or determine quantitatively constituents that are finely divided or dispersed through particles of aggregate (Fig. 4).

Ordinarily, analysis is performed on at least three size fractions of coarse aggregate and five size fractions of the fine aggregate. The results may be used to compute the petrographic composition of the aggregate in any gradation comprising the analyzed fractions. Occasionally, one analysis may be performed on a graded aggregate. For such an analysis, the composition by count usually differs greatly from the composition by weight.

Numerical results of the analysis may be expressed by weight or count for particles retained on the No. 30 sieve, but for the finer fractions, the results are based only upon count of grains unless a correction factor is applied. Consequently, consistent analyses of all fractions of fine and coarse aggregate can be reported only by count. Analysis by count is the more appropriate technically because the influence of particles of given type upon performance of concrete depends primarily upon their frequency and distribution in the mass. However, when applied to coarse aggregate only, the petrographic analysis is more rapid if the relative proportion of the several rock types or facies is determined by weight.

Details of calculating and reporting are summarized in ASTM Recommended Practice C 295.

Observations Included in the Petrographic Examination:

In reporting the results of the petrographic examination, the petrographer should supply information on the following subjects as necessary to evaluation of the aggregate:

- Mineralogic and lithologic composition
- Particle shape
- Surface texture
- Internal fracturing
- Coatings
- Porosity, permeability, and absorption
- Volume change, softening and disintegration with wetting or wetting and drying
- Thermal properties
- Strength and elasticity
- Density
- Hardness
- Chemical activity
  - Solubility
  - Oxidation, hydration, and carbonation
  - Alkali-silica reactivity
  - Alkali-carbonate reactivity
  - Sulfates
  - Sulfides
  - Cation exchange reactions
  - Organic substances

The significance of these properties is discussed by Rhoades and Mielenz [*7,8*], Swenson and Chaly [*9*], Hansen

TABLE 1—EXAMPLE OF TABULATION OF A PETROGRAPHIC ANALYSIS OF GRAVEL.
__________ Plant, __________ Company, Near Denver, Colo.
Sample No. ________

Rock Types	Size Fraction, per cent by weight[a]			Description of Rock Types	Physical Quality	Chemical Quality
	1½ to ¾ in.	¾ to ⅜ in.	⅜ to 3/16 in.			
Granite	29.5	40.0	48.6	medium- to fine-grained, rounded to fragmental	satisfactory	innocuous
Weathered granite	12.0	17.7	17.2	fractured, weathered, rounded to fragmental	fair	innocuous
Deeply weathered granite	...	0.5	...	fractured, slightly friable, rounded to fragmental	poor	innocuous
Coarse-grained granite	6.4	6.1	8.4	pink, rounded, includes some free quartz	satisfactory	innocuous
Fractured coarse-grained granite	...	0.9	...	pink, rounded, includes some free quartz	fair	innocuous
Rhyolite porphyry	0.8	0.2	1.1	microcrystalline, porphyritic, white to brown	satisfactory	innocuous
Andesite porphyry	2.2	1.2	0.1	microcrystalline, porphyritic, tan to green	satisfactory	innocuous
Weathered andesite porphyry	...	0.3	...	as above, fractured and weathered	fair	innocuous
Basalt	0.2	...	0.6	weathered, fractured, black, microcrystalline	fair	innocuous
Diorite	0.4	0.4	0.1	medium- to fine-grained, hard, massive	satisfactory	innocuous
Granite gneiss	32.2	14.3	15.8	hard, banded, fine- to medium-grained	satisfactory	innocuous
Weathered gneiss	10.3	7.8	2.7	as above, fractured to slightly friable	fair	innocuous
Deeply weathered gneiss	0.2	...	...	as above, intensely fractured to friable	poor	innocuous
Schist	2.2	2.3	2.2	hornblende schists, hard, rounded	satisfactory	innocuous
Fractured schist	...	0.4	0.2	as above, fractured	fair	innocuous
Quartzite	2.8	6.2	2.0	fine-grained, hard, massive to schistose	satisfactory	innocuous
Milky quartz	0.6	1.0	0.5	massive, hard, brittle, dense, smooth	fair	innocuous
Quartzose sandstone	...	0.1	...	fine-grained, massive, firm to hard	satisfactory	innocuous
Ferruginous sandstone	...	0.4	...	porous, brown, platy, quartzose	fair	innocuous
Shale	...	0.2	0.5	soft, absorptive, rounded, gray	poor	innocuous
Rhyolite	0.2	...	...	cryptocrystalline, porphyritic, pink to gray	satisfactory	deleterious

[a] Based upon analysis of 19.0 lb of 1½ to ¾-in., 2.7 lb of ¾ to ⅜-in., and 0.80 lb of ⅜ to 3/16-in. aggregate.

[*11*], and Mielenz [*10,12*]. A simple test to detect forms of pyrite that are likely to oxidize while enclosed in concrete was devised by Midgley [*13*] and is discussed more generally by Mielenz [*12*]. Petrographic criteria useful in detecting dolomitic rocks that are potentially susceptible of a deleterious degree of alkali-carbonate reactivity are described by Hadley [*14*] and by Gillott [*15*].

It is intended that the properties indicated above be determined qualitatively by observation of the mineralogic composition and texture of the particles or by simple tests, if appropriate. If other test data are available or if the particles can be compared petrographically with previously tested materials, the properties can be evaluated semiquantitatively or quantitatively.

TABLE 2—EXAMPLE OF TABULATION OF A PETROGRAPHIC ANALYSIS OF NATURAL SAND.

__________ Plant, __________ Company near Denver, Colo.
Sample No. ________

Constituents	Amount, as number of particles in per cent												
	In size fractions indicated[a]							In whole sample[b]					
	No. 4–8	No. 8–16	No. 16–30	No. 30–50	No. 50–100	No. 100–200	Passing No. 200	Physical quality			Chemical quality		
								S[c]	F[c]	P[c]	I[c]	D[c]	T[c]
Granite and granite gneiss	34.9	33.6	21.0	4.8	...	...	...	13.5	1.0	0.6	15.1	...	15.1
Pegmatite	34.2	28.7	2.0	...	...	...	...	8.2	0.8	0.2	9.2	...	9.2
Rhyolite tuff	0.8	0.9	0.3	...	...	...	...	0.3	...	...	...	0.3	0.3
Basalt	...	1.0	...	...	...	...	...	0.2	...	...	0.2	...	0.2
Sericite schist	2.4	0.7	...	9.5	11.2	1.1	...	4.7	...	...	4.7	...	4.7
Quartz and quartzite	24.6	41.2	59.2	65.0	43.0	48.8	28.8	51.6	...	...	51.6	...	51.6
Feldspar	2.6	3.3	16.7	11.1	18.7	23.8	10.5	...	11.4	...	11.4	...	11.4
Claystone	0.5	...	...	...	...	1.2	...	...	...	0.2	0.2	...	0.2
Chalcedonic chert	...	0.6	...	3.3	...	...	...	1.2	...	...	...	1.2	1.2
Mica	...	...	0.8	6.3	10.2	8.3	51.1	...	3.8	...	3.8	...	3.8
Hornblende, garnet, zircon, etc	...	...	...	...	16.9	16.8	9.6	2.3	...	...	2.3	...	2.3
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	82.0	17.0	1.0	98.5	1.5	100.0

[a] Based on count of 500 particles in each sieve fraction.

[b] Based on gradation of the sample received, and on the distribution of constituents by size fractions shown at the left above.

[c] S = satisfactory; F = fair; P = poor; I = chemically innocuous; D = potentially chemically deleterious; T = total of constituent in whole sample.

Condition of the Particles:

The following classification of properties is useful in cataloguing the physical and chemical condition of particles constituting an aggregate. Physical condition is defined by three terms: (1) satisfactory, (2) fair, and (3) poor; chemical stability in concrete is designated by two terms: (1) innocuous and (2) deleterious.

Satisfactory—Particles are hard to firm and relatively free from fractures, capillary absorption is very small or absent, and the surface texture is relatively rough.

Fair—Particles exhibit one or two of the following qualities: firm to friable, moderately fractured, capillary absorption small to moderate, surface relatively smooth and impermeable, very low compressibility, coefficient of thermal expansion approaching zero or being negative in one or more directions.

Poor—Particles exhibit one or more of the following qualities: friable to pulverulent, slake when wetted and dried, highly fractured, capillary absorption high, marked volume change with wetting and drying, combine three or more qualities indicated under "fair."

Innocuous—Particles contain no constituents which will dissolve or react chemically to a significant extent with constituents of the atmosphere, water, or hydrating portland cement while enclosed in concrete or mortar under ordinary conditions.

TABLE 3—EXAMPLE OF TABULATION OF A PETROGRAPHIC ANALYSIS OF EXPANDED CLAY AGGREGATE.

__________ Plant, __________ Company
Sample No. ________

Constituent	Amount, as number of particles in per cent									Remarks
	In size fractions indicated[a]								In whole sample[b]	
	¾ to ⅜ in.	⅜ to 3/16 in.	No. 4–8	No. 8–16	No. 16–30	No. 30–50	No. 50–100	Passing No. 100		
Black to gray, vesicular particles	44.6	30.2	36.2	44.1	46.5	55.0	62.2	64.4	42.4	vesicular, hard
Black to gray, vesicular particles	13.1	1.7	...	...	...	...	...	...	1.2	vesicular, friable
Red to tan, vesicular particles	26.1	29.5	20.2	25.0	15.4	24.0	21.8	20.6	23.5	vesicular, hard
Red to tan, vesicular particles	7.7	1.8	...	...	...	...	...	...	0.8	vesicular, friable
Red to brown, bricklike particles	4.9	23.6	32.3	22.0	25.6	12.4	7.8	4.9	21.3	not vesicular, firm to fragile
Gray to pink, bricklike particles	2.8	9.8	7.7	8.8	9.2	3.0	1.6	1.1	7.1	not vesicular, fragile, many slake in water
Gray to pink, bricklike particles	0.6	1.0	0.4	...	...	...	...	...	0.4	not vesicular, friable, many slake in water
Gray to black, friable particles	...	1.7	2.0	...	0.8	0.3	...	...	1.0	not vesicular, contain coal
Granite	0.2	0.4	0.4	...	1.4	2.8	4.7	7.0	1.4	hard, dense
Sandstone	...	0.2	...	...	...	...	...	...	0.1	hard, fine-grained
Coal	...	0.1	0.8	0.1	1.1	2.5	1.9	2.0	0.8	hard to friable
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	

[a] Based on count of 500 particles in each size fraction.

[b] Based on gradation of the sample received, and on the distribution of constituents by size fractions shown at left above.

Deleterious—Particles contain one or more constituents in significant proportion which are known to either react chemically under conditions ordinarily prevailing in portland-cement concrete or mortar in such a manner as to produce significant volume change, interfere with the normal course of hydration of portland cement, or supply substances which might produce harmful effects upon mortar or concrete.

Coatings should be reported and evaluated separately because coatings usually are confined to portions of a deposit and, for crushed stone, the nature and abundance of coatings varies with processing methods and equipment. Similarly, particle shape should be considered apart from other aspects of physical quality because particle shape commonly is subject to control or modification by processing of the aggregate. Unless otherwise defined by applicable specifications, particles whose length is more than five times their width should be designated as "elongated pieces," and those having a ratio of width to thickness greater than five should be designated as "flat pieces." The measurement should be made in accordance with definitions of these terms as they appear in ASTM Definition of Terms Relating to Concrete and Concrete Aggregates (C 125).

TABLE 4—EXAMPLE OF TABULATION OF A PETROGRAPHIC ANALYSIS OF CRUSHED STONE COARSE AGGREGATE.[a]

____________ Plant, ____________ Company
Sample No. ________

Rock Type or Facies	Description of the Rock Type or Facies	Amount, per cent by weight[b]					
		Physical quality			Chemical quality		
		S[c]	F[c]	P[c]	I[c]	D[c]	T[c]
Dense dolomitic limestone	gray to buff, contains sparse organic matter with pyrite and marcasite	56.2	...	...	56.2	...	56.2
Soft dolomitic limestone	gray to buff, soft to friable, slightly porous, sparse organic matter with pyrite and marcasite	...	33.9	...	33.9	...	33.9
Soft, organic dolomitic limestone	same as above except containing one or more seams of iron sulfides and organic matter	...	4.2	...	4.2	...	4.2
Limestone	white to gray, coarse- to medium-grained	2.4	...	...	2.4	...	2.4
Laminated limestone	laminated, fine-grained, iron sulfides and organic matter abundant	...	2.1	...	2.1	...	2.1
Chalcedonic limestone	white to gray, particles of zones of chalcedony evident	1.0	...	...	...	1.0	1.0
Chalcedonic chert	conchoidal fracture, dense	...	0.1	...	...	0.1	0.1
Sandstone	includes also grains of quartz and feldspar	0.1	...	...	0.1	...	0.1
Total		59.7	40.3	...	98.9	1.1	100.0

[a] Sample graded in accordance with specifications of the ____________ State Highway Dept. for 1½ in. to No. 4 aggregate for concrete highway pavement.

[b] Based upon analysis of 25.9 lb of aggregate split from the sample.

[c] S = satisfactory; F = fair; P = poor; I = not deleteriously alkali reactive; D = potentially deleteriously alkali reactive; T = total of constituent in the sample.

Summarizing the Petrographic Examination:

Tables 1–4 exemplify a variety of forms in which the petrographic analysis may be reported. All are based upon samples received as a part of engineering investigations. The tabulations always are accompanied by appropriate discussion and supplementary description. Table 1 is in the form generally employed by the Bureau of Reclamation [*16*]. It departs somewhat from the format recommended in ASTM Recommended Practice C 295, but the inclusion of pertinent description facilitates interpretation of the analysis. The tabulation could be simplified by cataloguing the rock types into major and secondary classifications, such as "granite: fresh, moderately weathered or fractured, deeply weathered." The summary of physical and chemical quality is included in a separate tabulation.

Table 2 is the analysis of the sand produced with the gravel whose composition is summarized in Table 1. The format accords with ASTM Recommended Practice C 295. Table 3 is similar except that the designations of quality are not used because they are inappropriate for description of lightweight aggregate.

Table 4 is an analysis of a sample representing a commercial crushed stone coarse aggregate passing the 1½-in. sieve. The analysis was obtained because the aggregate apparently delayed or prevented development of specified strength by the concrete under certain conditions.

Courtesy of the Michigan State Highway Dept.
Observation of natural disintegration forewarns of possible difficulty in service.

FIG. 5—Cobbles of argillaceous limestone disrupted by freezing-thawing in the deposit, near Charlesvoix, Mich.

In the tabulation, the denotation of "innocuous" and "deleterious" is restricted to potential deleterious alkali reactivity because the significance of the sulfides and organic matter in the stone could not be evaluated.

PETROGRAPHIC EXAMINATION OF NATURAL AGGREGATES

Examination of Natural Aggregates in the Field:

Sand and gravel result from weathering and natural abrasion and impacting of rock and the deposition of the resulting particles along streams, in lakes or marine basins, or by wind or glaciers on the earth's surface. Consequently, sand and gravel are more or less complex mixtures of different kinds of rocks and minerals. Moreover, deposits of sand and gravel usually vary vertically by stratification and laterally because of the lenticular nature of zones or strata. The concrete-making qualities of the aggregate are influenced by these changes. Examination in the field also should reveal the variability of the sand and gravel with reference to unsound or deleterious particles, clay, and organic matter.

Deposits of sand and gravel commonly are changed by deposition of mineral matter from ground water or by weathering of the particles. Examination in the field should indicate the lateral and vertical extent and the physical nature of the coatings. Areas of the deposit free from coatings and zones so heavily coated as to preclude processing should be delineated.

Weathering of gravel and sand after formation of the deposit is common on terraces and at lower levels of deposits along existing stream channels. The examination should indicate the extent and distribution of such weathering.

Close observation of gravel and sand exposed on the surface of the deposit commonly will reveal unsound particles which slake or fracture with freezing-thawing or wetting-drying (Fig. 5). Identification of such particles will aid evaluation of the petrographic examination performed in the laboratory. In interpreting the effects of natural freezing and thawing, the consequences of particle size should be evaluated [*17*]. Representative specimens of particles affected by freezing and thawing should be packaged separately and transmitted to the laboratory with samples of the gravel and sand. Water-soluble salts in coatings or ground water also may be revealed by efflorescence at or near the surface of the deposit. Their presence forewarns of the need for quantitative determination of their concentration in the aggregate.

Key:

- — Section of river carrying reactive sand or gravel
- × Source of reactive crushed stone
- ● Structure affected by alkali-silica reaction
- ○ Structure affected by alkali-carbonate reaction

FIG. 6—Location of structures in the United States known to be affected by alkali-aggregate reaction in concrete, sources of sand or gravel subject to alkali-silica reaction, and sources of crushed stone subject to alkali-carbonate reaction.

Examination of Natural Aggregates in the Laboratory:

Samples and data from the field should be examined to determine: (1) the abundance of individual lithologic or mineralogic types; (2) the abundance of particles in various physical conditions and degrees of chemical reactivity; (3) the composition, frequency, abundance, and physical nature of coatings; (4) particle shape; and (5) in a qualitative way, the possible contributions of particles of the several types to properties of concrete (Tables 1 and 2).

Natural aggregates may contain more than 20 rock and mineral types. Consequently, petrographic examination commonly is time consuming. Based upon the similarity in composition and probable performance in concrete, two or more rock types commonly can be combined into a single category with considerable saving in time and without loss in validity of the analysis. For example, granites, quartz monzonites, and granite gneisses—or rhyolites, dacites, and latites—of similar composition and physical condition might be combined, thus eliminating the need for tedious examination sufficient to effect a separation.

Soft and altered particles may be original constituents of sand and gravel, or may be developed by weathering in the deposit. Weathering in the deposit is especially significant because the alteration affects most or all particles, causing softening and absorptivity in the superficial portion of the particles. This action decreases both the bond with cement and the strength and durability of the concrete.

Coatings on gravel and sand vary from minute spots and films to a cement which produces zones of sandstone or conglomerate in the deposit. Coatings usually are composed of silt, clay, and calcium carbonate; but organic matter, iron oxides, opal, manganiferous substances, alkali and alkali - earth sulfates, and soluble phosphates have been identified [*8*,*18*]. The petrographic examination should reveal the composition, abundance, physical properties, probable potential chemical reactivity, and the ease with which the coatings are removed by impact and abrasion.

Courtesy of the Bureau of Reclamation, U.S. Department of Interior

Observation of such disintegration indicates unsoundness of stone in at least portions of a quarry.

FIG. 7—Disintegration of argillaceous facies of limestone used as riprap at Chickamauga Dam, Tenn.

Potentially alkali reactive gravel and sand occur along many important rivers in the United States and have been responsible for serious distress in many concrete structures (Fig. 6). The location of many known deposits of alkali reactive natural aggregate in western United States has been reported by Holland and Cook [*19*]. The known alkali reactive substances are the silica minerals, opal, chalcedony, tridymite, and cristobalite; glassy to cryptocrystalline rhyolites, dacites, latites, and andesites, and their tuffs; at least certain artificial siliceous glasses; and least certain phyllites. Alkali-reactive dolomitic rocks may occur as constituents of natural aggregates. Any rock containing a significant proportion of these substances is potentially deleteriously reactive.

PETROGRAPHIC EXAMINATION OF CRUSHED STONE

Examination of Stone in the Field:

Rock formations are massive or stratified; the strata can occur in any attitude with respect to the horizontal; and rock may vary widely in porosity, hardness, toughness, degree of fracturing or chemical reactivity. For example, the alkali reactivity of dolomitic limestones typically varies widely even within the same quarry when deleterious facies are present [*14*,*15*].

Rock formations commonly contain zones of faulting, jointing, or local shearing, within which the materials are fractured or chemically decomposed. Certain zones may contain deleterious or unsound substances, such as chalcedonic or opaline chert and clay or shale in limestone or dolomite. Especially in warm humid areas, rock formations commonly are fractured, leached, and partially decomposed near the surface.

These features should be discovered by geologic and petrographic examination of natural exposures, quarried faces or other excavations, and drilled core. Petrographic examination in the field should include examination of exposures and of stone from the formation used in the area for fill, ballast, riprap, and masonry. This survey may reveal lithologic varieties which have failed in the natural exposure to freezing-thawing or wetting-drying or to oxidation and hydration (Fig. 7). The significance of size of fragments should be recognized in any evaluation of such observations [*17*]. Disintegration of rock on natural exposure commonly does not coincide with results of the sulfate soundness test [*20*]. Shale, claystone, and argillaceous rocks, including deeply altered basalts and diabases, may slake and fracture during brief exposure even though they appear sound when excavated. Soluble salts usually will be revealed by efflorescence at exposed surfaces.

Examination of Crushed Stone in the Laboratory:

As is indicated above, crushed stone aggregates commonly are complex petrographically. Consequently, they should be examined in the detail required for natural aggregate (Table 4).

Petrographic examination of stone in the form of quarried blocks or irregular pieces should include inspection of the entire sample. The examination should establish the relative abundance of individual rock types or facies (Fig. 8). Specimens representative of each type should be obtained by sawing or coring of typical pieces; these specimens serve for special tests, detailed petrographic examination, and reference.

If the sample of stone was submitted for crushing tests in the laboratory, the manufactured aggregate produced should be examined to determine the effects of natural fracturing, jointing, and internal

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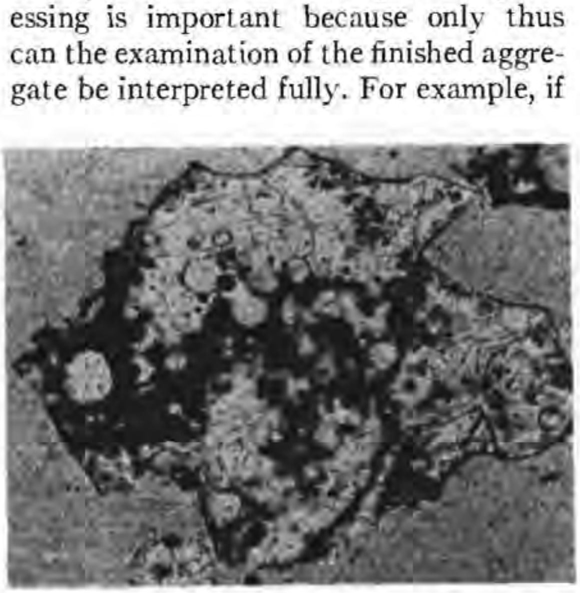
The physical quality was indicated as follows: satisfactory, 61 per cent; fair, 34 per cent; poor, 5 per cent by weight.

FIG. 8—Crushed limestone aggregate containing seams and pieces of illite shale at Webster Dam, Kans. (×$\frac{1}{2}$).

texture on particle size and shape; frequency of fractures and seams within the particles; distribution of unsound or deleterious substances in the size fractions; and the abundance and composition of crusher dust. These features should be correlated with the processing equipment and methods employed.

Inspection of the stone prior to processing is important because only thus can the examination of the finished aggregate be interpreted fully. For example, if unsound or deleterious particles constitute 10 per cent of the finished aggregate, was this proportion derived from approximately one piece in ten of the original sample, or does a typical piece of the stone contain approximately 10 per cent of unsound material? The former possibility suggests that the quarry should be examined to determine whether the unsound zones can be avoided or wasted; the latter suggests that the material should not be used as aggregate in permanent construction.



Courtesy of the Bureau of Reclamation, U.S. Department of Interior

The dark areas are concentrations of microcrystalline melilite and merwinite.

FIG. 9—Photomicrograph of granulated blast-furnace slag. Note the vesicles (bubbles) in the glass phase (white) (×80). Reduced one half for reproduction.

If the samples are in the form of drilled core, the entire length of core should be examined and compared with logs available from the driller and geologist. Especial attention must be given sections in which core loss was high or complete, inasmuch as such zones commonly represent fractured, altered, or otherwise unsound rock. The core should be examined by means of the hand lens, stereoscopic microscope, and petrographic microscope, as necessary, to establish variations in lithology; frequency and intensity of fracturing; content of clay and shale, regardless of rock type; and presence of deleterious substances, such as sulfides, soluble salts, and alkali reactive substances. These observations should be correlated from hole to hole so that the variation in lithology or quality of the rock, both in depth and laterally, is established.

The quality of the aggregate to be expected from the formation represented by the cores also will be indicated by petrographic examination of aggregate produced from cores in the laboratory.

PETROGRAPHIC EXAMINATION OF BLAST-FURNACE SLAG

Blast-Furnace Slag:

Blast-furnace slag is the nonmetallic product, consisting essentially of silicates and aluminosilicates of calcium and of other cations, which is developed simultaneously with iron in a blast furnace (see ASTM Specifications C 125). Three general types of blast-furnace slag are used for concrete aggregates, namely, air-cooled slag, granulated slag, and lightweight slag [*19*]. Petrographic examination of lightweight or expanded slag will be discussed in a later section.

Performance of the Petrographic Examination of Blast-Furnace Slag:

The procedure for petrographic examination of blast-furnace slag is not included specifically in ASTM Recommended Practice C 295. However, the

TABLE 5—COMPOUNDS OCCURRING IN BLAST-FURNACE SLAG.[a]

Compound	Chemical Formula	Compound	Chemical Formula
Gehlenite	$2CaO \cdot Al_2O_3 \cdot SiO_2$	oldhamite	CaS
Akermanite	$2CaO \cdot MgO \cdot 2SiO_2$	ferrous sulfide	FeS
Pseudowollastonite	$\alpha CaO \cdot SiO_2$	manganous sulfide	MnS
Wollastonite	$\beta CaO \cdot SiO_2$	spinel	$(Mg, Fe)O \cdot Al_2O_3$
Bredigite	$\alpha' 2CaO \cdot SiO_2$	anorthite	$CaO \cdot Al_2O_3 \cdot 2SiO_2$
Larnite	$\beta 2CaO \cdot SiO_2$	periclase	MgO
γ-dicalcium silicate	$\gamma 2CaO \cdot SiO_2$	lime	CaO
Olivine	$2(Mg, Fe)O \cdot SiO_2$		
Merwinite	$3CaO \cdot MgO \cdot 2SiO_2$	cristobalite	SiO_2
Rankinite	$3CaO \cdot 2SiO_2$	calcium aluminate	$CaO \cdot Al_2O_3$
Monticellite	$CaO \cdot MgO \cdot SiO_2$	cordierite	$2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$
Pyroxene			
Diopside	$CaO \cdot (Mg, Fe)O \cdot 2SiO_2$	sillimanite	$Al_2O_3 \cdot SiO_2$
Enstatite	$MgO \cdot SiO_2$	mullite	$3Al_2O_3 \cdot 2SiO_2$
Clinoenstatite	$MgO \cdot SiO_2$	madisonite	$2CaO \cdot 2MgO \cdot Al_2O_3 \cdot 3SiO_2$

[a] Compiled from several sources, primarily Nurse and Midgley [*23*], McCaffery et al [*29*], and American Concrete Inst. Committee 201 [*31*].

TABLE 6—MOST FREQUENTLY OCCURRING COMBINATIONS OF COMPOUNDS OF $CaO \cdot MgO \cdot Al_2O_3 \cdot SiO_2$ PRODUCED BY CRYSTALLIZATION OF BLAST-FURNACE SLAG.[a]

Flux Stone	Combination of Compounds[b]												
	C_2AS	C_2MS_2	C_2S	CS	C_3S_2	C_3MS_2	MA	CMS_2	CMS	CAS_2	M_2S	MS	MgO
Limestone	X	X	X		X								
	X	X	X			X							
	X	X				X	X						
	X	X					X			X			
	X	X		X						X			
Dolomite		X		X				X		X			
		X						X		X	X		
		X					X			X	X		
		X					X		X		X		
		X				X	X		X				
						X	X		X				X
								X		X	X	X	

[a] After Nurse and Midgley [*23*]

[b] Key: C_2AS = gehlenite } melilite
C_2MS_2 = akermanite } melilite
C_2S = dicalcium silicate
CS = wollastonite or pseudowollastonite
C_3S_2 = rankinite
C_3MS_2 = merwinite
MA = spinel
CMS_2 = diopside
CMS = monticellite
CAS_2 = anorthite
M_2S = forsterite
MS = enstatite
MgO = periclase

instructions provided for examination of ledge rock, crushed stone, and manufactured sand are applicable. In addition to the indicated microscopical methods, examination of polished and etched surfaces in reflected light is a valuable technique, being preferred by some petrographers because the surfaces are easy to prepare in sizes larger than thin sections, the two-dimensional aspect simplifies quantitative estimation of composition, and microchemical tests can be used to identify various phases in the section under study.

Air-cooled slag is more or less well crystallized, depending primarily upon the method of disposal employed at the steel plant. Such slag crushes to angular and approximately equidimensional pieces whose surface texture is pitted, rough, or conchoidal. Crystals range from submicroscopic to several millimeters in size. Abrasion resistance relates to glass content and the condition of internal stress [*21*]. Well-granulated slag is substantially all glass; crystals occur individually or in clusters scattered through the glass matrix. Incipient crystallization produces brown or opaque areas in thin section (Fig. 9).

The petrographic examination of blast-furnace slag aggregate should include description of the various types of slag as well as of contaminating substances. The slag constituent usually can be segregated into two or more varieties, depending upon particle shape, surface texture, color, vesicularity, crystallinity, or presence of products of weathering. Each type should be studied in some detail to assure identification of potentially deleterious compounds.

More than 20 compounds have been identified in blast-furnace slag (Table 5), but even well-crystallized slag rarely contains more than five compounds (Table 6). The typical constituent of blast-furnace slag is melilite, a compound of variable composition between akermanite ($2CaO \cdot MgO \cdot 2SiO_2$) and gehlenite ($2CaO \cdot Al_2O_3 \cdot SiO_2$). Pseudowollastonite and anorthite are of common occurrence. High-lime blast-furnace slags commonly contain one or more forms of calcium disilicate (α, β, or γ forms of $2CaO \cdot SiO_2$). Magnesian blast-furnace slags usually contain monticellite, forsterite, or merwinite. Calcium sulfide almost always is present in small proportion. Sulfides of manganous manganese and ferrous iron are common. Properties and techniques for identification of these compounds are summarized by Rigby [*22*], Nurse and Midgley [*23*], Insley and Frechette [*24*], and Snow [*25,26*], and in standard works on mineralogy. X-ray diffraction methods are necessary if crystalline phases are submicroscopic and are a great aid if a petrographer is developing experience independently in this field.

Several original constituents of blast-furnace slag may be deleterious to the performance of concrete. Sulfides released into the cement paste matrix produce innocuous greenish staining of the interior of concrete; occasionally, a mottled aspect is produced on surfaces of damp concrete [*12*]. Presence of colloidal sulfides is suggested by yellow or brown coloration of the glass phase [*21*]. Gypsum commonly forms in blast-furnace slag by weathering; the significance of such occurrences remains to be evaluated [*12*]. Inversion of β-dicalcium silicate to the γ-dicalcium silicate, with the accompanying 10 per cent increase in volume of the crystals, causes "dusting" or "blowing" of slag [*21,27*]. The inversion ordinarily takes place before the slag has cooled, and the disintegrated material is removed by screening in the production of coarse aggregate. In less severe occurrences the disintegration takes place slowly, producing pieces that are partly or wholly weak and friable, and thus unsuitable for concrete aggregate. This action of dicalcium silicate can be avoided by maintaining a ratio of CaO to SiO_2 in the slag sufficiently low to prevent formation of the compound, or by chilling the molten slag so that the compound does not crystallize [*21*]. If air-cooled slag is poured in thin layers, rapid cooling ordinarily arrests the compound in the β modification. Dicalcium silicate can be identified microscopically in slag by special techniques [*27*]. Scattered crystals of β-dicalcium silicate commonly are stable in blast-furnace slag and so are innocuous.

Free lime (CaO) and magnesia (MgO) are extremely rare as constituents of air-cooled blast-furnace slag and are not likely to form either as a primary phase or devitrification product in granulated, blast-furnace slag [*23*,*28*].[3] Nevertheless, their absence or presence and abundance should be established by the petrographic examination. These compounds are deleterious because of the increase in solid volume resulting from hydration or carbonation in place. Cristobalite has been reported as a constituent of blast-furnace slag [*29*]. This compound is potentially alkali reactive; if identified, its abundance should be determined. The glass phase of normal blast-furnace slag is not deleteriously reactive with cement alkalies.

Contaminating substances whose presence or absence should be established by petrographic examination are metallic iron, iron carbide, coke, and incompletely fused fluxstone. The last is important because delayed hydration and carbonation of free lime and magnesia may produce expansion of the concrete and popouts. Metallic iron and iron carbides rust by oxidation and hydration if exposed at the surface of concrete.

PETROGRAPHIC EXAMINATION OF LIGHTWEIGHT CONCRETE AGGREGATES

Expanded Clay, Shale, and Slate:

Petrographic examination of lightweight aggregate should include segregation of the particles into as many categories as required to adequately describe the sample (Table 3). Particles of expanded material may be segregated on the basis of particle shape, surface texture, development of a coating or "skin," vesicularity, and friability. Incompletely expanded particles and particles not expanded should be distinguished from vesicular ones. These should be separated on the bais of porosity, absorptivity, density, friability, softness, and reaction to water (softening, slaking, or swelling). The petrographic examination supplements standard tests in distinguishing clayey particles from "clay lumps" determined in accordance with ASTM Method C 142. Other materials to be identified and determined quantitatively are underburned or raw material, coal, and rock particles. The content of raw material commonly can be established most easily and accurately by X-ray diffraction or differential thermal analysis.

The individual types of particles also should be analyzed petrographically to establish the presence of free magnesia or lime. These result mainly from decomposition of calcium and magnesium carbonates in the feed during firing. They may produce distress or popouts in concrete or concrete products unless the aggregate is water- or steam-cured prior to use [*30*].

Petrographic examination also assists selection of raw materials, development of manufacturing methods and equipment, and process control.

Cinders:

Cinders used as concrete aggregate are the residue from high-temperature combustion of coal and coke in industrial furnaces. Petrographic examination should determine the physical nature of the cinder particles on the basis of composition, friability, softness, particle shape, and surface texture. Especial attention should be given to identification of sulfides, sulfates, coal, and coke.

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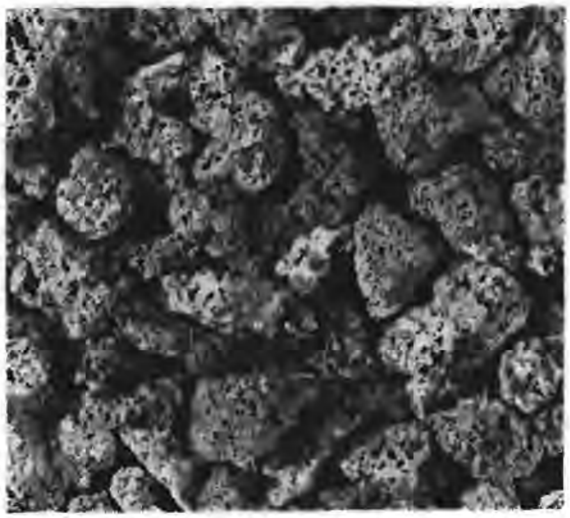
[3] Unlike blast-furnace slags, basic open-hearth slags commonly contain free oxides that are subject to hydration in portland-cement concrete, namely, free lime (CaO) and magnesiowüstite, a solid solution of MgO, MnO, and FeO

Expanded Blast-Furnace Slag:

Expanded blast-furnace slag is produced by carefully controlled intermingling of molten slag and water or steam in one of several ways [*21*]. The petrographic examination should describe the aggregate in terms of the nature of the expanded particles, including their particle shape, surface texture, friability, or softness, effects of weathering in stockpiles, and content of contaminating substances, such as dense slag.



Courtesy of the Bureau of Reclamation, U.S. Department of Interior

The pieces are black, gray, and reddish brown. Note the rounded vesicles.

FIG. 10—Typical basaltic scoria aggregate (natural size). Reduced one half for reproduction.

Pumice, Scoria, Tuff, and Volcanic Cinder:

Pumice, scoria, tuff, and volcanic cinder used for lightweight aggregate are naturally-occurring porous or vesicular volcanic materials. Pumice is a very highly porous and vesicular volcanic rock composed largely of natural glass drawn into approximately parallel or loosely entwined fibers and tubes. Scoria is a highly porous and vesicular volcanic rock in which the vesicles typically are rounded or elliptical in cross section, the interstitial glass occurring as thin films (Fig. 10). Tuff is a general term designating consolidated volcanic ash of any lithologic type or physical character. Volcanic cinder is a loose accumulation of highly vesicular (scoriaceous) fragments of lava, predominantly ranging from 4 to 32 mm in diameter.

Petrographic examination of these types of aggregate includes segregation of the particles on the basis of particle shape, surface texture, porosity or vesicularity, fracturing, friability or softness, weathering, specific gravity, secondary deposits in voids, coatings, and potential alkali reactivity. Extraneous or contaminating substances are primarily dense particles of volcanic rock and organic matter. In production of lightweight aggregate, two types of volcanic materials are intermixed occasionally for economy or to control gradation or unit weight. The type and relative proportion of the materials can be established by petrographic examination.

Volcanic glass with an index of refraction less than 1.535 is potentially deleteriously reactive with cement alkalies; glass whose index is in the range 1.535 to 1.570 probably is alkali reactive. Opal, chalcedony, tridymite, and cristobalite are also common alkali-reactive constituents of volcanic aggregates. However, in spite of alkali-aggregate reactions and formation of alkalic silica gel, expansion usually is prevented by the abundant voids into which the hydrating gel can escape without development of excessive stress in the mortar. For example, a very highly porous rhyolite tuff from Hideaway Park, Colo., containing abundant tridymite caused only 0.041 per cent expansion of a high-alkali cement mortar during one year of moist storage in accordance with ASTM Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227), yet the specimen contained abundant alkalic silica gel [*32*]. A similar but dense tuff from near Castle Rock, Colo., produced an expansion of 0.400 per cent under the same conditions.

Perlite:

When heated rapidly to fusion, certain obsidians and pitchstones release gases which, being trapped within the molten glass, vesiculate the rock, and cause disruption into small pieces. The product is known commercially as perlite.

Petrographic examination should indicate the composition of the aggregate in terms of particle shape, surface texture, composition, density, friability, or fragility, and potential alkali reactivity. Perlite may contain particles of dense volcanic rock or individual crystals.

Being composed of volcanic glass, typical perlite is potentially reactive with cement alkalies, although significant expansion may not occur because of the porosity of the particles. However, laboratory tests demonstrate that certain perlites produce significant expansion of mortar stored in accordance with ASTM Method C 227 in combination with either high-alkali or low-alkali cement [*33,34*]. Such volume change will not necessarily cause structural distress if appropriately accommodated in the design.

Exfoliated Vermiculite:

Exfoliated vermiculite is produced by rapid heating of the micaceous mineral, vermiculite. Release of combined water expands the crystals—like an accordion—increasing the volume to as much as 30 times its original size. The degree of expansion varies widely, depending upon mineralogic properties and purity and the conditions of firing.

During petrographic examination, the particles of vermiculite are segregated by degree of expansion, elasticity or brittleness of the flakes, and fragility of the expanded crystals. These differ significantly within individual samples from some sources, especially from marginal deposits where the vermiculite grades into hydrobiotite or biotite. Also to be reported is intermixture of the vermiculite with particles of rocks and minerals occurring with the vermiculite in the deposit.

Diatomite:

Crushed and sized natural diatomite typically is soft, porous, absorptive, and ranges from firm to pulverulent. Finely divided opal and opaline skeletons of diatoms are the predominant constituents. Fine sand, silt, clay, and volcanic ash are present in widely differing proportions. At least certain diatomites produce significant expansion of mortars stored in accordance with ASTM Method C 227, with both high- and low-alkali cement [*33,34*].

CONCLUSION

Petrographic examination should be included in the investigation and testing of concrete aggregate for use in permanent construction. Applied in the field, the method aids exploration and sampling and permits preliminary evaluation of materials from alternative sources. Detailed examination of aggregate in the laboratory supplements the standard acceptance tests, especially by: (1) detecting adverse properties, (2) comparing the aggregate with aggregates for which service records or previous tests are available, (3) explaining results of tests and justifying special tests as required, (4) detecting contamination, and (5) determining the efficiency and relative merit of processing and manufacturing methods.

Validity of the results depends upon the training and experience of the petrographer. However, with proper training and the adoption of uniform techniques and nomenclature, subjective elements in the examination are not significant.

The methods can be applied effectively to sand, gravel, crushed stone, slag, and natural or synthetic lightweight aggregate.

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Concrete Aggregates

GRADING AND SURFACE AREA

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This discussion will be limited to the effect of grading and maximum size of aggregate on the properties of concrete used in ordinary construction. No-slump, lightweight, and heavy concrete will not be considered, although much of the discussion is applicable to all types of concrete because aggregate, cement, water, and entrained air, of which concrete is composed, fit together as absolute volumes and in no other way.

Aggregate comprises about 55 per cent of the volume of mortar containing aggregate graded up to $\frac{3}{16}$-in.-max size and about 85 per cent of the volume of mass concrete containing aggregate graded up to 6-in.-max size. Thus, it is not surprising that the way the particles of aggregate fit together in the mix, as influenced by their gradation, shape, and surface texture, has an important effect on the workability and finishing characteristics of the fresh concrete and the properties of the hardened concrete. The influence of aggregate grading on the properties of concrete has been studied since the invention of portland cement, and many methods have been proposed for arriving at an "ideal" grading that would be applicable for all aggregates. None of these has been universally successful because of economic considerations, effect of particle shape and texture of the aggregate, and differences in cements from different mills [*1*].[2] Grading specifications have been developed, however, which on the average will produce a concrete of satisfactory properties from materials available in a particular area.

Historical Background

As early as 1845, W. H. Wright discussed the grading of aggregates in his "Treatise on Mortars" in which he states that sands of various sizes may be mixed together with great economy of cementing matter; the fine sands, in this case, fill up the interstices of the coarser kinds, and a greater density is obtained in the same volume [*2*]. This is at variance with the conclusions reached by the great French investigator, R. Feret, whose classical papers were published between 1890 and 1900 [*3*]. Feret concluded that the best mixture of sand and cement for mortar is made up of coarse and fine grains only, with no intermediate grains. Many since Feret have pointed out the advantages of "gaps" or "jump grading," the most recent being the Australian, L. Boyd Mercer, in a paper published in 1951 [*4*]. Feret also concluded that the grading of concrete aggregate had little effect on the compressive strength of mortars as seen in the following statement: "For all series of plastic mortars made with

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

the same cement and inert sands, the resistance to compression after the same time of set under identical conditions is solely a function of the ratio $C/(E + V)$ whatever may be the nature and size of the sand, the proportions of the elements —sand, cement, and water—of which each is composed." In the formula, E and V represent the volume of water and air, and C, the volume of cement.

In 1907, Fuller and Thompson published their celebrated paper, "The Laws of Proportioning Concrete" [*5*], in which they concluded that grading for denseness gives greatest strength, and that the grading curve of the best mixture resembles a parabola. In spite of the fact that many experimenters since then have found that grading affects the strength only indirectly, and that aggregate graded to produce maximum density produces extremely harsh concrete, credit is given these authors for the popularity of the smooth grading curve for aggregate which exists today. In 1916, Wig, Williams, and Gates concluded that there is no definite relation between the gradation of an aggregate and the compressive strength of the concrete and that the so-called maximum density curve (Fuller's curve) does not represent the curve for maximum density except for the particular materials used in the tests from which it was derived [*6*]. In 1923, Talbot and Richart agreed that aggregate graded to produce maximum density gave a harsh mixture that is very difficult to place, and that these minimum-voids gradings of aggregate, and even the next finer ones, cannot be considered usable gradings for ordinary concreting operations [*7*].

In 1918 Edwards [*8*] and in 1919 Young [*9*] proposed a method of proportioning based on the surface area of the aggregate to be wetted. Other things being equal, it was concluded that the concrete which is made from aggregate having the least surface area will require the least water in excess of that required to wet the cement and will consequently be the strongest. Abrams and others found that the surface area of the aggregate may vary widely without any appreciable difference in the concrete strength, and that water required to produce a given consistency is dependent to a large extent on the character of the aggregate apart from its surface area.

Abrams [*10*] roposed the "fineness modulus" for arriving at a satisfactory grading. He stated: "Any sieve analysis curve of aggregate which will give the same fineness modulus will require the same quantity of water to produce a mix of the same plasticity and gives concrete of the same strength, so long as it is not too coarse for the quantity of cement used." The fineness modulus is an index of the coarseness or fineness of an aggregate, but, because different gradings will give the same fineness modulus, it does not define the grading. The usefulness of the fineness modulus was extended by Swayze and Gruenwald [*11*] and Walker and Bartel [*12*] by including the cement in the calculations of the modulus.

Weymouth in 1933 and in subsequent publications proposed a "particle interference" method for arriving at a satisfactory grading [*13–15*]. He computed the volume relationships between successive size groups of particles based on the assumption that the particles of each group are distributed throughout the mass in such a way that the distance between them is equal to the mean diameter of the particles of the next smaller size group plus the thickness of the cement film between them. He stated that particle interference occurred between two successive sizes when the distance between particles is not sufficient to allow free passage of the smaller particles. The determination of gradings by the Weymouth method usually results in

SIEVE SIZE	PER CENT FINE AND COARSE SEPARATED			PER CENT FINE AND COARSE COMBINED		
	INDIVIDUAL	RETAINED	PASSING	INDIVIDUAL	RETAINED	PASSING
3 INCH						
1½ INCH	1	1	99	1	1	99
3/4 INCH	43	44	56	27	28	72
3/8 INCH	33	77	23	21	49	51
No. 4	23	100	0	14	63	37
No. 4	1	1	99	0	—	—
No. 8	14	15	85	5	68	32
No. 16	15	30	70	6	74	26
No. 30	25	55	45	9	83	17
No. 50	25	80	20	9	92	8
No. 100	18	98	2	7	99	1
PAN	2	100	0	1	100	0
FM		2.79				
PER CENT SAND			37			
SIEVE SIZE ARE BASED ON SQUARE OPENING						

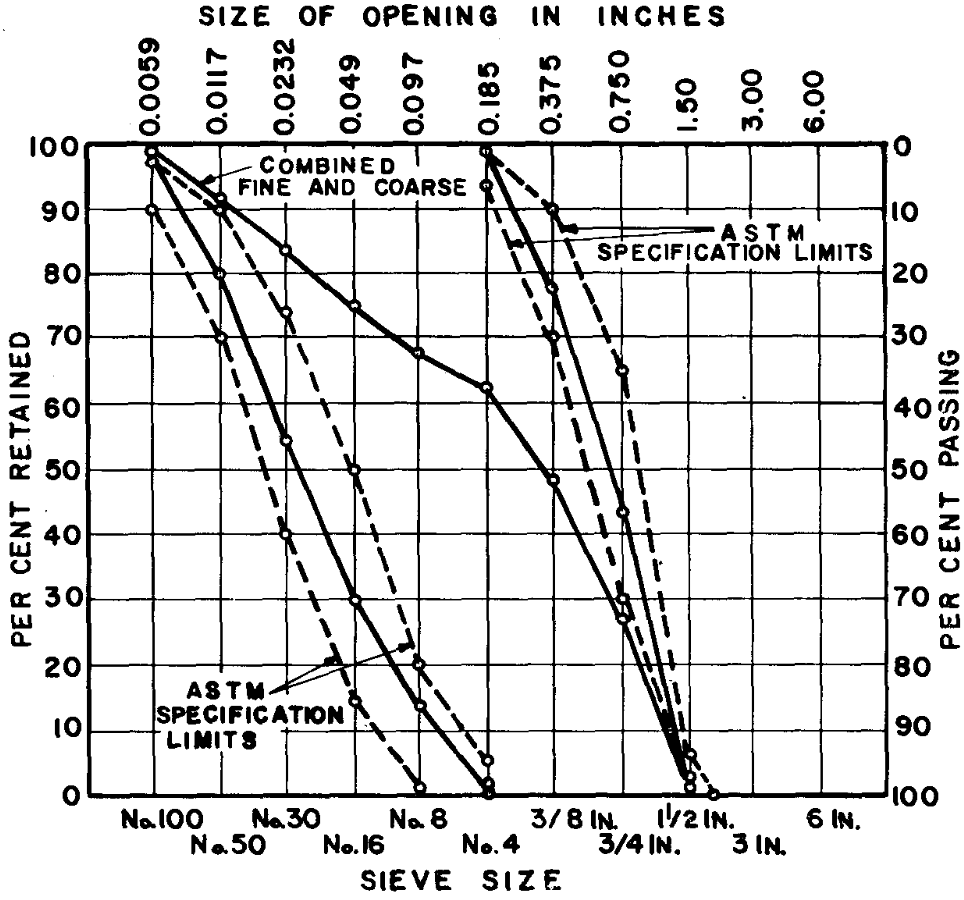

FIG. 1—Typical size distribution for aggregate graded up to 1½-in.-max size.

gradings finer than necessary for satisfactory workability.

Many other methods and formulas have been proposed for proportioning concrete and arriving at an optimum grading, but they only point up the fact that none is completely acceptable and that a reliable final selection can be made only by trial of the materials in a concrete mix. This is well stated by Mercer [*4*]: "It is undesirable to demand continuous gradings which will cause hardship or unduly increase the cost of the work, unless it has been established beyond doubt that such grading is essential. Grading specifications should be different for different localities and should take into account available local materials and conditions." Some of the modern grading specifications used by national and state organizations are shown in the tables included in a paper by the author on grading of mineral aggregates [*1*].

NOMENCLATURE

The particle size distribution of aggregate as determined by separation with standard sieves is known as its gradation. Sieve analysis, screen analysis, and mechanical analysis are terms used synonymously in referring to the gradation of aggregate.

A convenient system of expressing the gradation of aggregates is one in which the consecutive sieve openings are constantly doubled, such as $\frac{3}{8}$, $\frac{3}{4}$, and $1\frac{1}{2}$ in. Under such a system employing a logarithmic scale, lines can be spaced at constant intervals to represent the successive sizes. Figure 1 is an example of a chart drawn on this basis.

Gradings are expressed as the total percentage passing each sieve, the total percentage retained on each sieve, or the percentage retained between consecutive sieves. It makes little difference which method of expression is employed as long as it is understood. Generally it is best to conform to the custom of the area.

The fineness modulus shown in the table of Fig. 1 is used as a ready index of coarseness or fineness of the material. It is an empirical factor obtained by adding the cumulative percentages of the material retained on each of the particular sieves shown in Fig. 1 and dividing the sum by 100. Thus, the larger the figure, the coarser is the material. ASTM Definitions of Terms Relating to Concrete and Concrete Aggregates (C 125) define fineness modulus and other terms relating to concrete and concrete aggregate.

ASTM Definition of the Term Screen (Sieve) (E 13) defines a screen (sieve) as a plate or sheet or a woven cloth or other device with regularly spaced apertures of uniform size mounted in a suitable frame or holder for use in separating material according to size. A note indicates that in mechanical analysis testing work the term "sieve" applies to an apparatus in which the apertures are square and the term "screen" to an apparatus in which the apertures are circular. Specifications for sieves are given in ASTM Specifications for Sieve for Testing Purposes (Wire Cloth Sieves, Round-Hole and Square-Hole Plate Screens or Sieves) (E 11).

SIZE SEPARATION

It is common practice to separate aggregate into its component sizes for recombining in selected proportions in the concrete mix. Such separation is necessary for the production of uniform concrete, and it has been found to be economical for any sizable job, since the amount of cement saved more than offsets processing costs [*16*].

The average grading of a sand and gravel deposit in many cases is acceptable, but it is never uniform throughout, and it is not uncommon to have the percentage of sand range from 20 to 75 per cent from adjacent locations in the deposit. For concrete containing aggregate graded up to 1½-in.-max size, it would be impossible to produce a workable concrete free from segregation with 20 per cent sand, and 75 per cent sand would require about 2 sacks of cement above that required with a normal amount of sand to produce a desired strength. In other words, to make sure of a certain minimum strength with such a variation in sand percentages, the cement content would have to be maintained at 2 sacks above that required with a normal percentage of sand. The grading of the coarse aggregate also varies from one location to another in a deposit, and it is necessary to separate it into sizes to meet the requirements of ASTM Specifications for Concrete Aggregates (C 33). This separation for recombining in the mix pays dividends through the ease with which the resulting uniform workable mix can be placed and the saving in cement that is realized. As in the case of natural aggregates, it is desirable to separate manufactured aggregates into sizes, because the product from any one crusher extends over a wide range, and as many as four crushers may be used consecutively in reducing a rock or slag to the desired sizes.

Although many different gradings have been found to produce suitable concrete, the gradings cannot vary suddenly or widely during concreting operations because of their pronounced effect on the slump and workability of the concrete. Once a grading has been selected, it must be maintained within rather close limits for uniform workability and desired properties in the hardened concrete.

Uniformity of concrete is of such importance on large jobs that some specifications for these projects require that the grading of fine aggregate shall be controlled so that the fineness moduli of at least four of any five consecutive test samples of the fine aggregates, as delivered to the mixer, shall not vary more than 0.15 from the average fineness modulus of all samples taken during the first month's operation [*17*].

Perfect separation of aggregates cannot be accomplished at reasonable cost, and each size produced contains some undersize material. Specifications usually limit the amount of oversize and undersize permitted in any size fraction. It has been found that, where there is an excessive amount of either, segregation will occur in the stockpiling and batching operations, and slugs of fine or coarse material will reach the mixer. Because of the segregation which usually occurs in handling, it is desirable that the ratio of the size openings of the upper and lower screens which determine any size fraction be not greater than 2 to 1. This practice is generally followed except for the finest size of coarse aggregate where the ratio is usually increased above the 2 to 1 figure, and in this fraction it is common practice to extend the size from $\frac{3}{16}$ in. to ¾ or 1 in. Segregation in such finer size fractions may be very pronounced, with portions of the material to reach the mixer containing a preponderance of fines below the ⅜-in. size. Under such conditions it is difficult to obtain uniformly workable concrete. The condition is further aggravated by breakage and segregation which occurs in the larger sizes of coarse aggregate during stockpiling and handling. Lenses of fines which form in large stockpiles are another constant source of trouble. Breakage and segregation can be minimized through careful and proper handling of the aggregate. Some have found it economical to finish-screen the aggregate just before it goes into the batching bins, and where this is done, acceptable separation can be assured.

MAXIMUM SIZE AGGREGATE

Table 1 shows the amount of mortar (water, cement, sand, and entrained air) required, on the average, to produce a workable concrete with aggregates graded up to the maximum sizes listed.

For a given set of materials, the strength and other properties of concrete are governed more by the ratio of water to cement used and the amount of air entrained than by other factors. However, concrete made with smaller maximum sizes of aggregates will produce higher strengths at the same water-cement ratio than concrete made with the larger maximum sizes of aggregate of the same quality. Nevertheless, because less water is required to produce a given slump with the larger maximum size aggregate less cement is required with the larger maximum size aggregate to produce a desired strength for concretes having compressive strengths below about 4000 psi. As the strength of the concrete is increased above the 4000 psi level, the maximum size aggregate which will give the most economical cement requirement from a strength standpoint decreases. In the range of high-strength concrete, because the quality, surface texture, and particle shape of the aggregate has a marked effect on the strength of concrete that can be obtained, it is necessary that tests be made to determine the optimum maximum size aggregate which will require the least amount of cement to produce the desired strength [*18*,*24*]. There is some evidence that smaller maximum sizes produce slightly higher flexural strengths [*19*,*22*].

TABLE 1—MORTAR REQUIREMENTS FOR WORKABLE CONCRETE WITH VARIOUS MAXIMUM AGGREGATE SIZES.

Maximum size aggregate........	3/16 in. (sand)	⅜ in.	½ in.	¾ in.	1 in.	1½ in.	2 in.	3 in.	6 in.
NONAIR-ENTRAINED CONCRETE									
Water, lb per cubic yd[a]....	465	385	365	340	325	300	285	270	235
Cement, sacks per cubic yd[b]	9¼	7¾	7⅓	6¾	6½	6	5¾	5	4¾
Fine aggregate, per cent[c]...	100	62	54	49	44	40	37	34	28
Entrapped air, per cent[d]....	6	3	2.5	2	1.5	1	0.5	0.3	0.2
AIR-ENTRAINED CONCRETE									
Water, lb per cubic yd.....	415	340	325	300	285	265	250	235	200
Cement, lb per cubic yd....	8⅓	6¾	6½	6	5¾	5⅓	5	4¾	4
Fine aggregate, per cent....	100	58	50	45	40	37	34	31	25
Total air, per cent[e]........	13	8	7	6	5	4.5	4	3.5	3

[a] Approximate amount of mixing water in pounds per cubic yard of concrete required for 3-in slump with reasonably well-shaped angular coarse aggregate.

[b] Cement required in sacks per cubic yard of concrete for 0.53 water-cement ratio, 6 gal of water per sack.

[c] Approximate percentage of fine aggregate of total aggregate by absolute volume.

[d] Approximate percentage by volume of air entrapped in nonair-entrained concrete.

[e] Recommended average total percentage of air required for frost resistance, from Table 3, Recommended Practice for Selecting Proportions for Concrete, Am. Concrete Inst., 1954.

For the same water-cement ratio, compressive strengths of air-entrained concretes for the recommended percentages of total air listed in Table 1 are about 20 per cent less than for comparable nonair-entrained concrete.

For a given concrete, drying shrinkage decreases as the amount of mixing water is decreased; also, in massive structures it is desirable to maintain the cement content of the mix at a minimum to reduce the volume change resulting from the temperature rise caused by the heat of hydration of the cement [*20*]. In such massive structures in which concrete of strengths below 4000 psi is required, it is desirable to use aggregate graded up to the largest practicable maximum size.

The maximum size aggregate that can be used in a mix is limited by the type and thickness of the section to be constructed, the amount and spacing of reinforcement, the mixing and placing equipment, and the availability of the aggregate in the desired sizes [*21*]. The maximum size should not be larger than one fifth of the narrowest dimension between sides of forms, nor larger than three fourths of the minimum clear spacing between reinforcing bars. Table 2, taken from the Recommended Practice for Selecting Proportions for Concrete of the American Concrete Inst., gives the maximum sizes of aggregate recommended for various types of construction.[3]

TABLE 2—MAXIMUM SIZES OF AGGREGATE FOR VARIOUS TYPES OF CONSTRUCTION.

Minimum Dimension of Section, in.	Maximum Size of Aggregate, Based on Square Openings, in.			
	Reinforced Walls, Beams, and Columns	Unreinforced Walls	Heavily Reinforced Slabs	Lightly Reinforced or Unreinforced Slabs
2½ to 5	½ to ¾	¾	¾ to 1	¾ to 1½
6 to 11	¾ to 1½	1½	1½	1½ to 3
12 to 29	1½ to 3	3	1½ to 3	3
30 or more	1½ to 3	6	1½ to 3	3 to 6

TABLE 3—SOME FINE AGGREGATE GRADINGS OUTSIDE ASTM SPECIFICATIONS WHICH PRODUCED ADEQUATE CONCRETE.

Sieve Size	Percentage Passing					
	ASTM Specifications C 33	Falcon Dam[a]	Webster Dam[b]	Wu Sheh Dam[c]	Gateway Tunnel[d]	Norris Dam[e]
⅜ in.	100	100	100	100	100	100
No. 4	95 to 100	100	100	100	100	100
No. 8	80 to 100	59	96	72	91	77
No. 16	50 to 85	35	86	47	82	51
No. 30	25 to 60	30	61	22	70	31
No. 50	10 to 30	28	13	6	30	20
No. 100	2 to 10	20	1	2	7	11
Fineness modulus		3.28	2.43	3.51	2.20	3.10

[a] Sand used in Falcon Dam, lower Rio Grande River, Tex.
[b] Sand used in Webster Dam, South Fork of Solomon River, Kans.
[c] Sand used in Wu Sheh Dam, Formosa.
[d] Sand used in Gateway Tunnel, Webster Basin Project, Utah.
[e] A manufactured sand. (The other four sands were natural river-worn material.)
NOTE—All concretes contained entrained air except Norris Dam.

Grading of Fine Aggregate:

The grading of fine aggregate has a much greater effect on workability of concrete than does the grading of the coarse aggregate. If the mortar is workable and satisfactory in other respects, it is usually possible to secure workable concrete by using enough mortar to fill the voids among the coarse aggregate particles and separate them so that

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[3] Recommended Practice for Selecting Proportions for Concrete, Am. Concrete Inst., 1954.

there is ample room for them to move in the mortar without interference. Experience has shown that usually very coarse sand or very fine sand is unsatisfactory for concrete mixtures. The coarse sand results in harshness, bleeding, and segregation; and the fine sand requires a comparatively large amount of water to produce the necessary fluidity and also tends to cause segregation. Fine aggregate gradings falling within the specification limits of ASTM Specifications C 33, shown in Table 3, should be satisfactory for most concretes.

Under these specifications the minimum percentage of material passing the Nos. 50 and 100 sieves may be reduced to 5 and 0, respectively, if the aggregate is to be used in air-entrained concrete containing more than 4½ bags of cement per cubic yard, or in nonair-entrained concrete containing more than 5½ bags of cement per cubic yard, or if an approved mineral admixture is used to supply the deficiency in fines passing these sieves. ASTM Specifications C 33 also require that fine aggregate shall have not more than 45 per cent retained between any two consecutive sieves of those shown in the table, and that the fineness modulus shall be not less than 2.3 nor more than 3.1.

Fine aggregates having gradings outside the C 33 specifications have produced satisfactory concrete in some instances, but where the behavior of an available sand having a grading falling outside accepted limits is unknown, and where the size of the job does not warrant the expense of trial mixes before specifications are written, it is recommended that an accepted specification such as C 33 be used. On large jobs involving many thousands of cubic yards of concrete, it is recommended that the average grading as produced from the deposit or quarry without blending or wasting to bring it within some predetermined limits be tried in trial mixes to determine whether the aggregate can be used without expensive processing. It is stressed, however, as has been discussed under Size Separation, that regardless of the grading selected it must be maintained within certain limits without sudden variations if satisfactory economical concrete is to be produced.

Table 3 lists five fine-aggregate gradings, each of which does not meet C 33 specifications in some respect; yet, very satisfactory workable concrete was produced with the first four gradings in air-entrained concrete. The Norris Dam fine aggregate was a crushed dolomite and gave satisfactory workability without entrained air.

In the case of Falcon, Wu Sheh, and Norris Dams, because of the large quantities of concrete involved, it was possible to investigate the aggregate and concrete prior to writing the specifications and permit gradings approaching those shown. In the case of Webster Dam and Gateway Tunnel, where the quantities of concrete were relatively small, grading data on the source of aggregate used were not available until after the contract was let, and the usual Bureau of Reclamation grading limits were specified.

It has already been mentioned that ASTM Specifications C 33 permit a reduction in the minimum amount of material passing the Nos. 50 and 100 sieves from that shown in Table 3 in the case of rich or air-entrained mixes. On the other hand, increased percentages of these sizes may be desirable in the case of the lean concrete used in mass construction.

In general, specifications permit more fines in manufactured sand than in natural sand. In the construction of Norris Dam, in which manufactured fine and coarse aggregates were used, it was found that a deficiency between the

Nos. 100 and 28 sieves could not be made up at a reasonable cost. It was determined that the effect of this deficiency—that is, poor workability and excessive "water gain"—could be minimized by using additional material finer than No. 100 but from which particles finer than No. 325 had been removed [*22*].

It is sometimes difficult to finish a floor because of the grading of the sand which causes "chattering" or waves to form under the trowel. In one case this "chattering" was eliminated by increasing the amount of material passing the No. 50 sieve from 12 to 18 per cent and the amount passing the No. 200 sieve from 1 to 4 per cent.

Coarse Aggregate:

As a rule it is less difficult to provide satisfactory grading in coarse aggregate than in sand. It is practicable to make size separation of the coarser material by screening at comparatively small expense and to recombine the separated sizes in desired proportions. Table 2 of ASTM Specifications C 33 shows the approximate ranges over which it will usually be practicable to vary the relative proportions of size fractions of coarse aggregate for concretes having various maximum sizes of aggregates and for optimum sand percentages. The combination of available materials which requires the least amount of mixing water for the conditions of placing and workability is the one that should be used.

AIR ENTRAINMENT AND GRADING

Since 1938, when it was realized that purposefully entrained air in concrete materially improves the resistance of concrete to frost action, improves its workability, and reduces segregation in the mix, its use has gradually increased until now it is required by many organizations for all types of concrete. The effect of the finely dispersed air bubbles obtained through the use of air-entraining agents is entirely different from that of the larger air voids which result from poorly graded aggregates; the larger voids do not improve the quality of the concrete and contribute to poor workability and segregation. Table 1 shows the reduction in fine-aggregate percentages which may be realized through the use of air entrainment in concrete. In addition to a reduction in the amount of fine aggregate which may be accomplished for a given set of materials and grading, it is sometimes possible to use a coarser or finer grading or a jump grading and obtain concrete of satisfactory workability with the proper amounts of entrained air. ASTM Specifications C 33 recognize the effect of entrained air on workability and permit a smaller percentage of fines for air-entrained concrete. Table 3 of this paper lists a number of fine-aggregate gradings which produced workable concrete with entrained air but were unsatisfactory without entrained air. In general, more poorly graded aggregates can be used for air-entrained concrete, but it is recommended where such aggregates are being considered that they be tried in trial mixes before the usual specifications are relaxed or changed. As the amount of fines—cement, mineral admixture, or fines in aggregate—is increased, the amount of air entrained for a given amount of air-entraining agent is decreased. This is of little consequence, however, because the desired amount of air can usually be obtained by increasing or decreasing the amount of air-entraining agent used.

CONCLUSION

Many methods have been proposed for arriving at an "ideal" grading, but none have been accepted universally because of economic considerations, particle shape and texture of the aggregate, or differences in portland cements. Grading affects the strength of concrete indirectly through its effect on the amount of water required to produce a plastic mix. On the other hand, particle shape and surface texture of the aggregate may have an appreciable effect on strength apart from what indirect effect they may have on the water requirement of the mix. Coarse, fine, and gap gradings have produced satisfactorily workable concrete under certain conditions, but unless trial mixes are made beforehand to determine whether they can be used, it is recommended that gradings be required to meet a specification such as ASTM Specification C 33. Economical, uniform concrete cannot be produced with pit-run or crusher-run aggregate, and it is necessary that the aggregate be separated into its component sizes so that it can be combined in the concrete mix within the limits of variation permitted by the specifications.

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STP169A-EB/Apr. 1966

Concrete Aggregates

SHAPE, SURFACE TEXTURE, AND COATINGS

By Bryant Mather,[1] Personal Member, ASTM

A property of aggregate is "significant" from the viewpoint of this publication only if it has a significant effect on the behavior of portland-cement concrete in service. Similarly, a test of aggregate is "significant" only if it yields useful information about a significant property or group of properties. The behavior of concrete in service is determined by the interaction of certain of its properties and the significant characteristics of the exposure to which it is subjected. Variations in shape and surface texture of, or the presence of coatings on, aggregate particles may significantly affect properties of concrete that are important to its behavior in service.

No ASTM methods exist by which quantitative determinations of particle shape, surface texture, or coatings can be made. Few satisfactory data are available concerning the relations between variation in these properties of aggregates and behavior of concrete. These properties have not been adequately defined, and adequate definitions are necessary before satisfactory information can be obtained regarding the degree to which aggregates differ with respect to them. The nature and degree of differences among aggregates with respect to these properties must be satisfactorily determined before their effect on the behavior of concrete can be properly evaluated.

The absence of satisfactory data is not due to failure of previous writers to suggest the probable significance of these properties. These properties are mentioned or discussed in the 1943 ASTM "Report on Significance of Tests of Concrete and Concrete Aggregates" in the papers by Gilkey [*1*],[2] Hubbard [*2*], Kriege [*3*], and Lang [*4*]; and in the 1948 ASTM Symposium on Mineral Aggregates in the papers by Allen [*5*], Rhoades andMielenz [*6*], Rockwood [*7*], Sweet [*8*], and Woolf [*9*]. Reports of work done in Australia, Denmark, France, Germany, Great Britain, Japan, Sweden, Switzerland, and the United States are cited in the list of references at the end of this paper. Standardized test methods have been developed by Markwick [*10,11*] and Shergold [*12,13*] of the Road Research Laboratory in Great Britain; by Schiel [*14–16*], Walz [*17*], Schulz [*18–20*], Schulze [*21*], Pickel and Rothfuchs [*22*], Pickel [*23*], and the Forschungsgesellschaft für das Strassenwesen [*24*], in Germany; by von Matern [*25*] in Sweden; by Feret [*26,27*] in France; and by Goldbeck [*28*], Huang [*29*], and the Corps of Engineers [*30–32*] in the United States.

The most careful studies of concepts and definitions of particle shape and surface texture have been made by

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

sedimentary petrologists, notably Wentworth [*33–35*], Wadell [*36–38*], Zingg [*39*], Krumbein [*40–42*], Pettijohn [*43*], and Sneed and Folk [*44*]. However, the relation of their work to aggregates for portland-cement concrete has received little attention, especially in the United States.

Two relatively independent properties, *sphericity* and *roundness*, control *particle shape*. *Sphericity* is the property that measures, depends upon, or varies with the ratio of the surface area of the particle to its volume, the relative lengths of its principal axes or those of the circumscribing rectangular prism, the relative settling velocity, and the ratio of the volume of the particle to that of the circumscribing sphere. *Roundness* is the property the measure of which depends upon the relative sharpness or angularity of the edges and corners of the particle.

Surface texture is the property the measure of which depends upon the relative degree to which particle surfaces are polished or dull, smooth or rough, and the type of roughness.

Coatings are layers of material, covering part or all of the surface of the particle, that have been deposited on the particle after its formation. Coatings usually, but not necessarily, consist of materials differing physically or chemically, or in both respects, from those composing the particle, and are not derived from the particle itself.

Sphericity, roundness or angularity, smoothness or roughness, and coatings of a particle are the result of the interaction of the nature, structure, and texture of the rock (or other material, for example, slag) of which the particle consists and the forces to which it was subjected during and after its formation. Rocks having closely spaced partings or cleavages in one or two directions tend to yield flat or elongated particles of low sphericity [*6*]. The design and operation of crushing equipment influence sphericity of crushed particles; generally, the greater the reduction ratio, the lower the sphericity [*7,10,45–47*]. Roundness is primarily a function of the strength and abrasion resistance of the material and the amount of wear to which the particle has been subjected. Surface texture depends on hardness, grain size, pore structure, and texture of the rock, and the degree to which forces acting on the particle surfaces have smoothed or roughened it. Hard, dense, fine-grained materials will generally have smooth fracture surfaces. Soft, loosely bonded coatings include clay coatings on inadequately scrubbed gravel particles and dust coatings on crushed particles. Hard, tightly bonded coatings are usually formed by precipitation of materials carried in solution in groundwater on sand and gravel particles in natural deposits.

The ASTM Recommended Practice for Petrographic Examination of Aggregates for Concrete (C 295) notes that such examinations are made to determine the physical and chemical properties of the material that may be observed by petrographic methods and that bear on the quality of the material for its intended use, and to describe and classify the constituents of the sample. It directs that pieces of coarse aggregate be examined to establish whether coatings are present. If they are, it should be determined whether the coatings consist of materials likely to be deleterious in concrete (opal, gypsum, easily soluble salts, organic matter) and how firmly the coatings are bonded to the pieces. The relevant features of each rock type found in the sample may include particle shape, particle surface, grain size, texture, and structure, including observations of pore space, packing of grains, and cementation of grains. This recommended practice points to the desirability of getting information on these properties but does not provide procedures for obtaining such information nor does it define the properties.

MEASUREMENT OF PROPERTIES

Sphericity:

Since 1942, the ASTM (C 125) Definitions of Terms Relating to Concrete and Concrete Aggregates has included the following statements:

"**Flat Piece**—One in which the ratio of the width to thickness of its circumscribing rectangular prism is greater than a specified value."

"**Elongated Piece**—One in which the ratio of the length to width of its circumscribing rectangular prism is greater than a specified value."

Since these statements provide no "specified value," they are descriptions of procedures for defining rather than definitions. The property with which they deal is sphericity. Sphericity was defined by Wadell [*36–38*] as the cube root of the ratio of the volume of the particle to the volume of the circumscribing sphere. If d equals the nominal diameter of a particle, that is, the diameter of a sphere of the same volume as the particle, and a equals the long dimension of the particle, that is, the diameter of the circumscribing sphere, then

$$\text{Sphericity} = \sqrt[3]{\frac{(\pi/6)d^3}{(\pi/6)a^3}} = \sqrt[3]{\frac{d^3}{a^3}} = \frac{d}{a}$$

Krumbein [*41*] pointed out that for most particles d^3 is approximately equal to abc, the product of the longest, intermediate, and shortest axes of the particle. Therefore,

$$\text{Sphericity} = \sqrt[3]{\frac{(\pi/6)abc}{(\pi/6)a^3}} = \sqrt[3]{\frac{bc}{a^2}}$$

$$\left(\frac{b}{a}\right)^2 = \frac{\text{Sphericity}^3}{c/b}$$

The ratios b/a and c/b were used by Zingg [*39*] in developing a sphericity classification that closely approximates that stipulated in ASTM Designation C 125 which uses the ratios L/W $(= a/b)$ and W/T $(=b/c)$.[3] If 1.5 is taken as the "specified value" in the statements in ASTM Designation C 125, the resulting definitions will yield the four following sphericity classes proposed by Zingg.

Classes Proposed by Zingg				Based on ASTM Designation C 125 Using 1.5 as the "Specified Value"		
Class	Name	b/a	c/b	Name	L/W	W/T
I	Disks (oblate)	$>2/3$	$<2/3$	flat	<1.5	>1.5
II	Spherical (equiaxial)	$>2/3$	$>2/3$	neither flat nor elongated	<1.5	<1.5
III	Blades (triaxial)	$<2/3$	$<2/3$	both flat and elongated	>1.5	>1.5
IV	Rods (prolate)	$<2/3$	$>2/3$	elongated	>1.5	<1.5

Figure 1 shows that each class as developed by Zingg and stipulated in ASTM Designation C 125, using 1.5 as the "specified value," includes particles of widely differing sphericity and that sphericity alone fails to differentiate the kinds of departure of the particle from spherical. The dashed lines in Fig. 1 drawn at the ratios of $\frac{1}{3}$ and 3, defining the stippled area, indicate the classes set up by the Corps of Engineers test method [*31*] based on the use of 3 as the specified value. Walz [*17*] proposed a plot indicating four classes based on L/T as abscissa and W/T as ordinate.

Sneed and Folk [*44*] found from a study of 1150 particles from the Lower Colorado River that the frequency distribu-

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[3] L, W, and T refer to length, width, and thickness; $L > W > T$.

tion for the ratio of *L-W/L-T* was essentially normal, indicating that a majority of the particles tended to have widths equal to about the mean of the length and thickness.

Plum [*48*] suggested expressing the degree of content of flat and elongated particles by the ratios *a/b* and *c/b*, respectively, where *a* is minimum thickness, *c* is maximum length, and *b* is mean size. Markwick [*10*] devised the procedure used in British Standard 812 [*49*] based on classifying as flat (flaky) those particles having a thickness less than 0.6 times the mean sieve size, and as elongated (long) those with a length greater than 1.8 times the mean sieve size. The weights of material passing the respective gages expressed as a percentage of the sample are designated "flakiness index" and "elongation index" [*12*]. Lees [*50*] reviewed the concepts and procedures of British Standard 812, 1960 [*48*] and concluded that the British Standard flakiness and elongation gages do not satisfactorily perform the function for which they were intended. He favored the use of caliper measurements. He recommended a return to Zingg's [*39*] ratio of 0.66 for both elongation and flatness-ratio boundaries between shape categories. AASHO Designation M 80 [*51*] does not differentiate flat and elongated particles since its reference is to those having "length greater than 5 times the average thickness." Schiel [*14*] devised a formula involving specific gravity, settling velocity (a function of thickness), and sieving (a function of width), the results of which are expressed as values from 100 to about 70. Good particle shape = 100, cubical = >86.5, fairly cubical = 83.5 to 86.5, flaky = 80.5 to 83.5, and very flaky = <80.5.

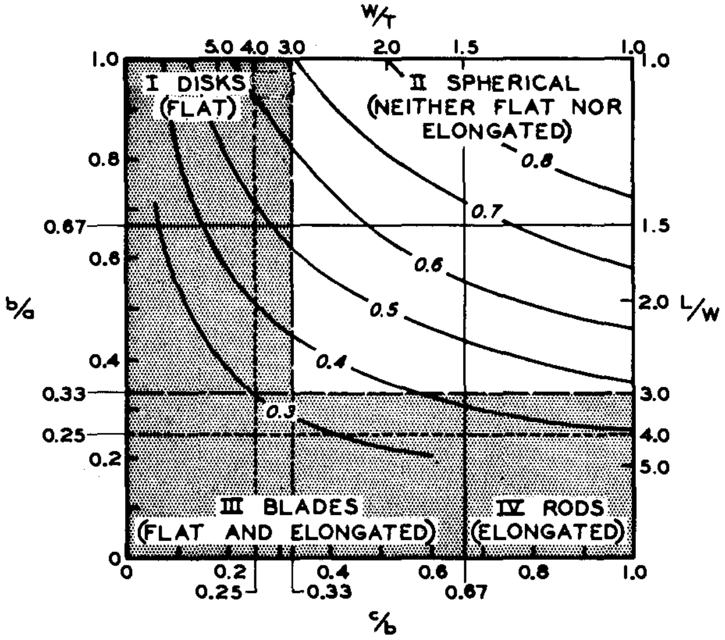


FIG. 1—Relation of sphericity (curves) calculated from $(b/a)^2 = (\text{sphericity}^3)/(c/b)$, Zingg Classes (I–IV), and ASTM C 125 Classes (*W/T*, *L/W*). After Krumbein [*41*, Fig. 4]. Stippled area indicates particles classed as flat, elongated, or both by the Corps of Engineers [*31*] using *W/T* and *L/W* = 3 as criteria.

Feret [*26,27*], Pickel [*23*], Pickel and Rothfuchs [*22*], and Stern [*52*] have used procedures based on the number of particles of a given sieve size that can be held in a container of a given volume. Plum [*48*] stated that the number of particles of desirable shape, when compacted, needed to fill a container of a given volume is smaller than the number needed if the particles are flat but greater than the number needed if they are elongated.

Huang [*29*] proposed a procedure for determining particle index using a rhombohedron-shaped mold into which aggregates are compacted first by 10 roddings and later by 50. Using the two unit weight values, two values for percentage of voids are calculated, and using these and a nomograph, a value for particle index is obtained. These values were compared with sphericity values calculated according to Wadell [*53*] with results shown in Fig. 2. Huang states that particle index takes account of shape (= sphericity), angularity, and surface texture. His work was related to soil-aggregate mixtures. Rex and Peck [*54*] proposed a method for evaluating simultaneously the effects of "shape" and "surface texture" by observing the rate at which sand of a single size fraction will run through a $\frac{3}{8}$-in. orifice. The rate for a given sand divided by that for the same size fraction of standard testing sand is the "time index."

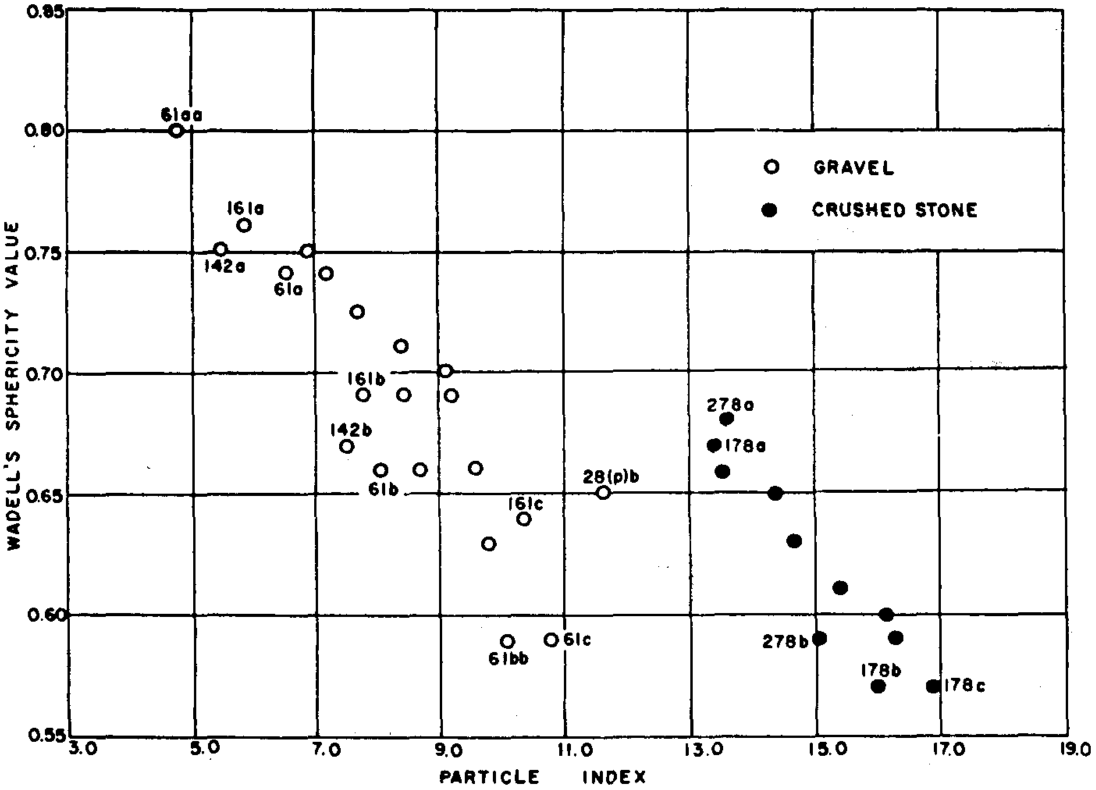


FIG. 2—Relation between particle index and Wadell's sphericity value of typical aggregate samples after [*29*].

The Corps of Engineers [*31*] and Schulze [*21*] have developed proportional calipers, or gages, to permit rapid determination of dimensional ratios of particles. These instruments operate so that as one of two jaws or slits is adjusted to a dimension of the particle, the other jaw or slit adjusts to a given submultiple thereof. Schulze's device (Fig. 3) involves two slits having a 1:3 ratio. The Corps of Engineers device (Fig. 4) has two jaws normally having a 1:3 ratio but capable of being set also at 1:2 or 1:5.

Schulze proposed setting the gage for length and rejecting the particle if its width is less than one third of its length. The Corps of Engineers procedure involves classification on criteria of both W/T and $L/W = 3$. A particle is classed as flat if the ratio of width to thickness is greater than 3 and elongated if the ratio of length to width is greater than 3.

Heywood [*55*] discussed a method developed by Hogan that is applicable to large particles. Surface area is calculated from the weight of the wax coating produced by immersing the particle in molten paraffin at a prescribed temperature. The sphericity of sand particles has also been studied by determinations of surface area by Davies and Rees [*56*] using the air permeability method suggested by Robertson and Emodi [*57*] based on the work of Carman [*58*] and Lea and Nurse [*59*].

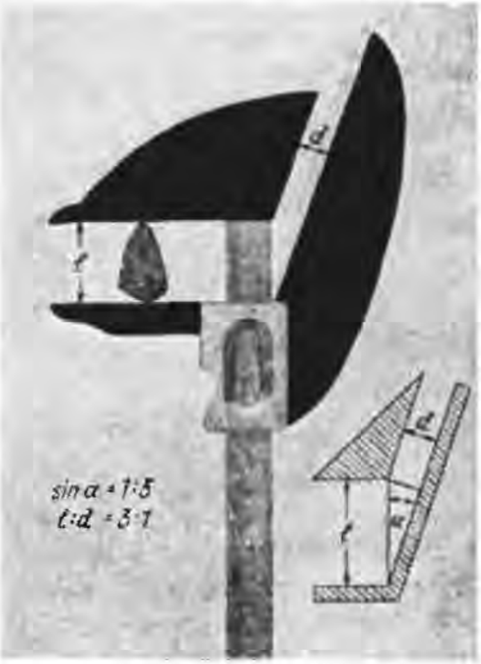


FIG. 3—Caliper, or gage, as developed by Schulze [*24*].

Roundness:

Roundness can be defined as the ratio of the average radius of curvature of the corners and edges of the particle to the radius of the maximum inscribed circle [*42*]. Numerical values of roundness are less frequently employed than are descriptive terms based on the following categories [*43*]:

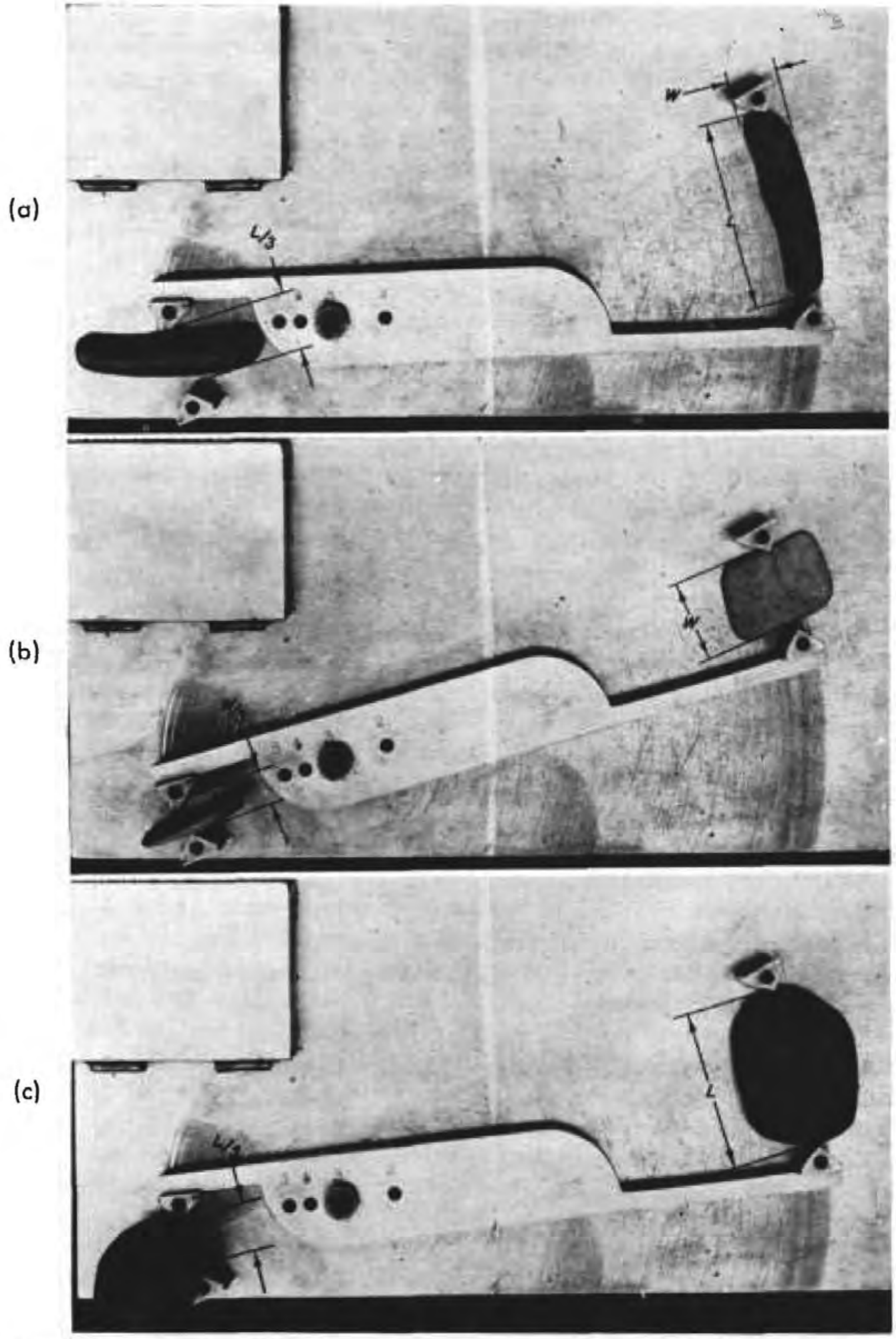
1. angular (little evidence of wear),
2. subangular (evidence of some wear, faces untouched),
3. subrounded (considerable wear, faces reduced in area),
4. rounded (faces almost gone), and
5. well rounded (no original faces).

Roundness is independent of sphericity. Data are not available to indicate the relation, if any, between empirical procedures designed to measure roundness (or angularity) of aggregate and the property, "roundness," as defined above. Shergold [*13*] suggested a procedure involving determination by a standard method of the percentage of voids in compacted samples of individual sieve fractions of coarse aggregate. He proposed the term "angularity number" = percentage voids − 33. The angularity number ranges from 0 for well-rounded gravel to eleven for very angular material. Goldbeck [*28*] described a similar method of determining voids in manufactured sands. Loudon [*60*] determined an "angularity factor" based on specific surface. Powers [*61*] pointed out that Loudon's angularity factor does not relate directly to angularity as defined here, but is rather merely the reciprocal of the sphericity factor. Mackey [*62*] reviewed various methods and recommended one based on particle outline. He assumed a shape of 1.0 for "the perfect ellipsoid" and made deductions for "irregularities."

Methods based on measurements of radii of curvature have been described by Wentworth [*34,35*] and Waddell [*36–38*].

Surface Texture:

Jones [*63*] suggested that a device such as the replica surface analyzer described



(*a*) *Test for elongation*—Right jaw of caliper is set equal to length L of particle; left jaw is then automatically set to $L/3$. Particle is tested to see if its width W is less or greater than $L/3$. W for this particle is $< L/3$; hence, it is "elongated" by the Corps of Engineers criterion.

(*b*) *Test for flatness*—Right jaw of caliper is set equal to width W of particle; left jaw is then set to $W/3$. Particle is tested to see if its thickness T is less or greater than $W/3$; T for this particle is $< W/3$; hence, it is "flat" by the Corps of Engineers criterion.

(*c*) *Test for elongation*—Test is similar to (*a*) but particle is not elongated.

FIG. 4—Corps of Engineers caliper.

by Herschmann [*64*] might be of some value in the study of aggregate surface texture and cited various reports on surface texture, surface finish, and surface roughness [*65–68*]. Roughness of aggregate particle surfaces should probably be expressed in terms of arithmetic average deviation of the actual surface from the mean surface. Various types of equipment have been developed for use in the evaluation of metal surfaces. The December, 1954, issue of *Mechanical Engineering* describes devices developed by Brush Electronics Co. and General Electric Co. Other equipment is produced by the Micrometrical Div. of the Bendix Corp. Such determinations of the effect of aggregate surface textures as have been made have described the aggregates studied only in terms as "rough," "fairly rough," "smooth," and "very smooth." Kriege [*3*] reported that in certain limestones the degree of roughness was found to range from 1.3 to 3.0 times the surface measured from plane polished shapes, but he does not relate these differences to performance in concrete. Kramrisch [*69*] described a procedure in which a linear profile was prepared and examined and presented results of work on about 140 samples representing about 30 different types of rock.

In addition to the quantitative measurement of roughness, the types of roughness may be of importance. Pettijohn [*43*] listed the following varieties of roughness: furrowed, grooved, scratched, ridged, pitted, dented, striated, frosted, and etched. Blanks [*70*] differentiated the relative significance of undulatory and abrupt rugosity. Knight and Knight [*71*] listed these types of surface texture: glassy, smooth, granular, crystalline (fine, medium, coarse), pitted, honeycombed, and porous.

Coatings:

The presence of a coating, its nature, thickness, continuity, and degree of adhesion or tenacity, can best be established by petrographic examination. Blanks [*70*] listed the following types of coatings noted by the U.S. Bureau of Reclamation: "silt, clay, gypsum, impure carbonates of lime and magnesia, iron oxides, opal, manganiferous substances, soluble phosphates, or mixtures of these substances." These are the characteristic natural coatings; artificial coatings include asphalt and portland-cement paste.

EFFECTS AND SIGNIFICANCE OF PROPERTIES

Sphericity:

Equidimensional particles are generally preferred to flat or elongated particles for use as concrete aggregates because they present less surface area per unit volume and generally produce tighter packing when consolidated. Powers [*61*] wrote, "From some points of view, perfect sphericity is the ideal particle shape, for spheres have the smallest specific surface area for a given nominal size, and an aggregation of spheres has the least percentage of voids for a given grading." For these reasons, equidimensional particles of a given grading require a minimum of cement paste for a given degree of workability of concrete. The extent to which the undesirability attributed to effects of flat and elongated particles may have resulted from the use of gradings that work best with equidimensional particles is not known. Smith and Kidd [*72*] described procedures used in connection with the production and use of 2,000,000 tons of crushed stone (dolomite) aggregate in the U.S. projects on the St. Lawrence Seaway. They stated that the percentages of each size group of coarse aggregate were chosen to produce a combined grading agreeing as closely as practicable to an ideal grading computed from a modification of Talbot's formula:

$$P = \sqrt{\frac{d}{D}} = \left(\frac{d}{D}\right)^{0.5}$$

where P is the accumulative percentage passing a sieve of size d for an aggregate having a maximum size D. The modification consisted of using 0.38 rather than 0.5 as the exponent. Smith and Kidd stated that "an exponent of 0.3 would be used with angular flat and elongated crushed aggregate and 0.5 with spherically shaped natural gravels." They do not give data on the particle shape of the aggregates they used nor on the variation in particle shape with difference in particle size. This, however, does tend to confirm the supposition that if a grading suitable for relatively spherical particles is employed with particles that are highly nonspherical, the results may be expected to be less satisafctory than if a more appropriate grading had been employed.

When the strength of the material of which the particles are composed is greater than that of the matrix, and the bond between the particles and the matrix is well developed, it would be expected that a concrete made with spherical aggregate particles would have less strength than one made with nonspherical particles. Shergold's results [*13*] indicate that changing sphericity (from 20 to 40 per cent flat particles) had little effect on the percentage of voids, whereas changing roundness had a direct progressive effect.

Various investigations of the effects of flat and elongated particles have been reported [*47,73–76*]. Markwick [*10*] observed that "there is widespread unanimity of opinion" that "flaky and elongated material is unsatisfactory" but "so far as can be ascertained, there are no references in the relevant literature to actual failures attributed" to such material. Gilkey [*1*] stated that flat or elongated particles may be positively objectionable either because of the uneconomic grading that they cause or because of structural weakness introduced within the mass. Pramstig [*77*] described flat and angular particles as "unfavorable" since "these require a higher percent of cement and are detrimental to the workability of the concrete." He described "sliver segregators" that remove unfavorably shaped particles.

Blanks [*70*] said, "Flat or elongated particles . . . tend to decrease workability and thus to require more sand, cement, and water. They tend to reduce bulk weight and decrease compressive strength. If flat particles become oriented with their large faces in a horizontal position, bleed water will be entrapped under them, a condition which prevents the development of good bond." Scholer [*78*] regarded this latter phenomenon as likely to develop pores along which weathering may progress.

Mercer [*79*], on the other hand, found it practical to proportion concrete mixtures of adequate workability and a water-cement ratio of 0.6 by weight with aggregates of which 50 to 90 per cent were flat or elongated particles or both. He, therefore, concludes that specification limits on particle shape are unduly restrictive, and notes that "there is good reason for preferring 'poor shape' whenever the controlling features are bond between aggregate particles and paste, and the modulus of rupture."

In another study, Mercer [*80*] showed that compressive strength can be materially reduced when flat particles of coarse aggregate are present in large numbers and oriented vertically. Tests of cylinders containing hand-placed flat particles that were then grouted gave the following results:

Condition	Compressive Strength, 7 days, psi	Unit Weight, lb/ft^3
Stone vertical	1330	159.0
Stone horizontal	2235	159.5
Mortar grout	2255	136.0
Normal arrangement	2260	160.0

Studies by Lane and Carlson [*81*] of the movement of gravel bed material in canals developed data on the relation of sphericity of gravel particles to movement that may be applicable to the effects of sphericity on workability of concrete. The effect of sphericity on susceptibility to movement by flowing water as contact load was found to be:

Sphericity Range	Relative Weights of Particles of Equal Susceptibility to Movement
0.5 to 0.6........	0.63
0.6 to 0.7........	0.67
0.7 to 0.8........	1.25
0.8 to 0.9........	1.67

Elongated particles have also been reported [*5*] to have had objectionable effects in pavement concrete by interfering with finishing operations; such effects were not found in the studies discussed by Walker [*76*].

Gilkey [*82*] reported tests in which concrete was made with broken scraps of plate glass crushed to pass a 1¼-in. sieve. Concrete made with ⅛-in. glass had lower compressive strength than that made with ¼-in. glass, which, in turn, had lower strength than that made with normal aggregate. He concluded that flat pieces are a source of weakness because they are loaded as beams. In other fields their effects may be of more significance, for example, a limit of 10 per cent was placed on flat and elongated pieces in specifications for trickling filter media [*83*]. This was based on ratios of 1.5:1 for L/W and W/T and, hence, corresponds to the classification of Zingg [*39*] (see Fig. 1). Lees [*84*] reviewed the parameters of particle shape and the methods of measurement. He concluded that for some mixtures containing asphalt as a binder a flaky aggregate has superior performance.

Roundness:

Almost all studies in which roundness was a variable have compared the performance of "rounded" gravel with "angular" crushed stone without indicating the degree to which roundness varied or the degree to which sphericity, surface texture, and other properties also varied. Well-rounded particles might be expected to require less cement paste for equal workability than angular particles of equal sphericity and similar surface texture. Shergold [*13*] showed that decreasing roundness or increasing angularity directly affects the percentage of voids in aggregate; this in turn affects workability or mixture proportions in concrete.

Blanks [*45*] reported experience on two similar projects where sand particles were in one case "well-rounded" and in the other "harsh and angular." He states:

> For concrete with 1½-in. max size and a water-cement ratio of 0.50, by weight, using the angular material, 300 lb of water and 6.39 sacks of cement per cubic yard of concrete were required. Using the rounded aggregate, the same factors were 220 lb of water and 4.69 sacks of cement per cubic yard of concrete . . . The latter concrete, in addition to being of higher quality by reason of its lower water requirement, saved 1.7 sacks of cement per cubic yard, which would more than pay for an effective abrading operation to improve the characteristics of the angular aggregate.

Malhotra [*85*] using the method proposed by Rex and Peck [*54*] studied nine natural sands and seven crushed sands; the time indexes ranged from 1.05 for Leighton Buzzard sand to 1.57 for a fine aggregate produced by crushing a coarse granite. He found a significant relation between flow of mortar and time index; sand with a higher time index had a higher water requirement.

Shergold [*13*] provided a procedure by which "angularity number" may be determined and notes that it was developed for use in connection with the British

Standard for Concrete Aggregates (B.S. 882:1944, Appendix M) which distinguishes "rounded" from "angular" but gives no method of defining or measuring roundness. Knight and Knight [71] noted that, in the case of sedimentary rocks, angularity indicates high quality, whereas rounded grains usually indicate a relatively low structural strength.

Surface Texture:

As surface smoothness increases, contact area decreases; hence, a highly polished particle will have less bonding area with the matrix than will a rough particle of the same volume. A smooth particle, however, will require a thinner layer of paste to lubricate its movement with respect to other aggregate particles. It will, therefore, permit a tighter packing for equal workability and, hence, will require a lower paste content than a rough particle of similar roundness and sphericity. Goldbeck [*86*] reported tests in which pieces of aggregate shaped to a 1-in.-square area were embedded in cement briquets and tested for bond strength in tension. The different surface textures and bond strengths in tension reported were:

Surface Texture of Aggregate	Bond Strength in Tension, psi		
	28 Days in Water	28 Days in Water, Then 28 Days in Air	28 Days in Water, Then 28 Cycles of Wetting and Drying
Rough, porous	350	260	235
Rough	240	275	230
Rough	215	300	245
Fairly rough	250	185	160
Smooth	120	45	...
Smooth, conchoidal	285	170	45
Very smooth	195	40	25

No specifications are known in which requirements are imposed for surface texture of aggregate particles.

Blanks [*70*] stated:

> Salients and depressions on the particles, particularly when the sides of these roughnesses are almost perpendicular to the general surface, assist the adherence of the paste to the aggregate. Undulatory roughness is less helpful and may even be harmful to bond as the mortar changes in volume. Rugosity is probably less significant than physical penetration of cement into the aggregate.

The beneficial effects of rough texture of aggregate on flexural strength were shown by some unpublished data developed by the Corps of Engineers for concrete made with a 5.5-bag-per-cubic-yard cement factor and a $1\frac{1}{2}$-in.-max size as follows:

Per Cent of Particles		Water-Cement Ratio, gal/bag	Strength, 28 days, psi	
Smooth	Rough		Flexural	Compressive
100	0	6.0	595	4850
50	50	6.4	645	4500
0	100	6.7	670	4125

Backstrom et al [*87*] reported studies in which eleven fine aggregates were divided into two groups: four having smooth surfaces and seven having rough surfaces based on examination using a microscope. Each sand was prepared to a given grading and used in concrete of a given water-cement ratio and comparable concentration of air-entraining admixture. The average values for surface area of voids were 742 in.$^{-1}$ for smooth and 1037 in.$^{-1}$ for rough fine aggregates, and the average void spacing factors were 0.0065 and 0.0045 in., respectively.

Coatings:

Coatings that are removed from aggregate particles during the mixing of the concrete have only the effect of altering the grading by adding the coating material to the mixture. Coatings that remain on the particles after the concrete has been placed affect the properties of the concrete exactly as if the materials of which they are composed were present other than as coatings. An opal coating or a gypsum coating may be undesirable because of its composition, but, if so, it is only incidental that it is a coating. Specifications for concrete aggregates exist in which "adherent coatings," "asphalt coatings," and "incrustations" or merely "coatings" are the basis for regarding the particles possessing them as deleterious or objectionable.

Lang [*4*] noted that adherent clayey coatings may interfere with bond, that nonadherent dust coatings will increase fines and water demand, and that little is known of the effects of cemented coatings. Goldbeck [*88*] reported that each 1 per cent of dust, probably as a consequence of its effect on water demand, will cause a 1 to 1½ per cent decrease in flexural strength and a 0 to 2 per cent decrease in compressive strength of concrete.

SPECIFICATIONS

ASTM Specifications for Concrete Aggregates (C 33) are silent regarding the properties discussed in this paper. The Federal Specifications [*89*] are similarly silent. The 1953 Corps of Engineers specifications for civil works [*90,91*] provided for an optional limit of 25 per cent on flat and elongated particles in coarse aggregate, as determined by the Corps of Engineers method [*31*], and instructed [*92*] that this requirement should be included in the specifications for a project when preliminary work indicated the need for so doing. In 1963 [*91,92*], however, this requirement had become mandatory for all work of sufficient importance to require use of the standard specifications. None of these specifications contain any specific requirements regarding surface texture and coatings. It, thus, appears that no cognizance is taken in specifications for general use in the United States of any of the properties of aggregates with which this paper is concerned except by the Corps of Engineers in its specification for concrete for civil works where flat and elongated particles, as defined by it, are limited to 25 per cent. I know of no case in which this requirement has caused rejection of a source of aggregates, and only one case in which the requirement caused a producer to modify his crushing procedures.

The British Standard for Single-Sized Gravel Aggregates for Roads [*93*] requires that the flakiness index not exceed 40 for 1½-in. nominal size aggregate and 35 for 1, ¾, and ½-in. nominal size aggregates. The determination is made as described by Markwick [*10*]. The British Standard for Concrete Aggregates from Natural Sources [*94*] requires that the aggregates be "free from adherent coatings such as clay" but does not require compliance with any specific limits relating to shape or surface texture. It also requires that the supplier shall provide, when requested by the purchaser, data on shape and surface texture as determined according to clause 10 of British Standard 812 [*49*] which are the methods described by Markwick [*10*].

CONCLUDING STATEMENT

Engineers who are required to evaluate the concrete-making properties of aggregates or to discuss the factors responsible for variations in the behavior of concrete are generally aware that variations in aggregate particle shape, surface texture, and coatings may produce effects of significance to these considerations. They have not been provided with adequate information with which to convert their general awareness of the potential significance of these variations into useful tools which they can employ in making engineering judgments. To provide such information, it will be necessary for scientists, engineers, and technologists concerned with these questions to: (1) develop adequate definitions and concepts of the properties, (2) develop suitable techniques for determining the degree to which aggregates differ with respect to them, and (3) determine the effects of such differences on the behavior of concrete. The results of such work may justify the use of aggregates that are now regarded as unsuitable, and may cause the establishment of new requirements for aggregates. In any case, the results of such work will contribute knowledge to an area in which there is now very little.

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Concrete Aggregates

WEIGHT, DENSITY, ABSORPTION, AND SURFACE MOISTURE

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Variations in the weight, density, absorption, and surface moisture of aggregates produce both independent and related effects on the properties of concrete. Some effects are the result of a direct relationship between one or more aggregate factors and a property of concrete, such as the influence of the weight and density of aggregates on the weight of concrete. Most of the important properties of concrete can also be influenced by these factors when they vary sufficiently to affect concrete proportions. Thus, an awareness of their significance and suitable methods of measurement can contribute to good control of concrete.

Unit Weight

Methods of Testing for Unit Weight:

The unit weight of a concrete aggregate is the weight of a unit volume of representative particles. In the United States, unit weight is usually expressed as the weight in pounds of 1 ft^3, and it is determined in accordance with ASTM Method of Test for Unit Weight of Aggregate (C 29), using $\frac{1}{10}$, $\frac{1}{3}$, $\frac{1}{2}$, or 1-ft^3 containers, depending on the nominal maximum size of the aggregate to be tested.

The method provides for both compacted and loose weight determinations. The compaction methods are usually employed in determining the unit weight of aggregates for use in the normal paving or structural types of concrete and for the very heavy aggregates used in concretes for special purposes. Aggregates having a maximum size of 2 in. or less are compacted by rodding, but a jigging procedure is specified for aggregates of a nominal maximum size greater than 2 in. but not exceeding 4 in.

A loose weight method of determining unit weight is used for lightweight aggregates of the types used in lightweight concrete products, lightweight structural concrete, and insulating concrete. Loose unit weight is determined by a shoveling procedure which is applicable to aggregates having a maximum size of 4 in. or less.

ASTM Method C 29 states that results on the same sample should check within 1 per cent. This degree of repeatability can readily be obtained with aggregates of 2-in. max size or less. However, the prescribed procedure for striking off samples of larger size aggregate by balancing aggregate projections against large voids below the top of the measure is conducive to such variations in sample weight that it is difficult to

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meet the 1 per cent limit for the large aggregates.

Hosking [*1*][3] has investigated the various factors that can affect the results of the unit weight test when different sizes of container are used. It is shown that the size of container selected, even though conforming to the size restrictions of ASTM Method C 29, can introduce significant variations in unit weight. The compacted unit weight of $\frac{1}{2}$ to $\frac{3}{8}$-in.-size material, for example, may vary as much as 2 lb/ft^3 depending on whether it is tested in a $\frac{1}{10}$ or $\frac{1}{2}$-ft^3 container. The effect of container size is even more pronounced for larger size aggregate and for uncompacted determinations. Differences due to container size are apparently related to boundary effects and the fact that the use of the same number of blows of the tamper for all sizes of container results in different compactive efforts per unit of area. Hosking recommends that the number of compactive blows be proportioned to the area of aggregate tamped and that the size of the container be related to the size of the aggregate tested.

A variation of the standard unit weight test was developed by Huang [*2*] in order to evaluate the geometric characteristics of shape, angularity, and surface texture of aggregates. In this test, both the voids resulting under a given condition of compaction and the rate of change in voids over a range of compactive effort are used to establish a "particle index" value. The geometric characteristics of polished aluminum spheres are used to provide a definite standard for comparing various kinds of aggregates. Huang, Auer, and Triffo [*3*] demonstrate that the particle index value can be used to measure the geometric characteristics of aggregates which affect the strength of soil aggregate mixtures and believe that it can be a useful tool in connection with other kinds of pavement mixtures involving aggregates.

Significance of Unit Weight Test:

The unit weight determination provides a method for classifying aggregates as lightweight, normal weight, or heavyweight. Such a classification serves to indicate whether the weight of the aggregate qualifies it as a suitable material for special uses. Unit weight is specified as a measure of quality of some aggregates and is used as a factor in some methods of designing concrete proportions. The uniformity of unit weight may also be of interest, either as a direct measure of quality or because of its influence on the control of concrete.

For many purposes, the significance of the unit weight determination depends on the fact that it reflects the void content of an assembly of particles. Void content is governed by the extent to which particles can be fitted together and, in addition to compactive effort, is influenced by their grading, shape, and to a lesser extent their surface texture. These are the same properties that determine the ease with which particles can be manipulated in concrete or, in other words, determine the workability of the concrete. Since concrete proportions are influenced by the workability of the mixture, it follows that the unit weight determination measures properties of aggregates which can affect the final quality of concrete.

The usefulness of void content of aggregates as a measure of angularity has long been recognized. Gray and Bell [*4*] have reported extensively on studies of void content of fine aggregates of varying angularity. They show that the workability and over-all quality of mortars are related to the percentage voids of the fine aggregate and suggest an upper limit of 53 per cent of voids as

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[3] The italic numbers in brackets refer to the list of references appended to this paper.

being adequate for preventing the use of materials having an objectionable particle shape such as stone screenings. The Virginia State Highway Dept., however, limits the void content of concrete sands to 50 per cent. In a study of gravels, Shergold [*5*] showed good agreement between an "angularity number," which equals the percentage voids − 33, and the workability of concrete as indicated by the compacting factor. Kaplan [*6*] used the angularity number to classify aggregates as to shape in a study designed to determine the effect of various aggregate properties on the strength of concrete. His work showed that approximately one quarter of the variation in strength could be related to differences in the shape of the aggregates. Bloem and Gaynor [*7*], in a somewhat similar study, were also able to relate shape and surface texture, as measured by void content, to the mixing water requirement and strength of concrete.

TABLE 1—UNIT WEIGHT REQUIREMENTS FOR LIGHTWEIGHT AGGREGATES FOR STRUCTURAL CONCRETE AND MASONRY UNITS.

Size Designation	Dry Loose Weight, max, lb ft^3
Fine aggregate	70
Coarse aggregate	55
Combined fine and coarse aggregate	65

TABLE 2—UNIT WEIGHT REQUIREMENT FOR LIGHTWEIGHT AGGREGATES FOR INSULATING CONCRETE.

Size Designation	Dry Loose Weight, lb/ft^3	
	Min	Max
Group I		
Perlite	7½	12
Vermiculite	6	10
Group II		
Fine aggregate	...	70
Coarse aggregate	...	55
Combined fine and coarse aggregate	...	65

In ASTM Specifications for Concrete Aggregate (C 33), the only reference to unit weight relates to blast-furnace slag, where the compacted weight when graded as it will be used in concrete is required to be not less than 70-lb/ft^3. This limiting value is based on extensive tests by Hubbard [*8*] which showed that blast furnace slag aggregates below 1½ in. in nominal maximum size and weighing less than 70 lb/ft^3 compacted produce concrete of lower strength than slags with unit weights in the range of 70 to 113 lb/ft^3.

In the case of lightweight materials, ASTM specifications use unit weight as a criterion for determining that an aggregate qualifies as a lightweight material and whether it is suitable for insulating concrete, structural concrete, or masonry units. The unit weight requirements which are stipulated in the ASTM Specifications for Lightweight Aggregates for Structural Concrete (C 330 – 64 T) and for Masonry Units (C 331 – 64 T) are given in Table 1.

The ASTM Specification for Lightweight Aggregates for Insulating Concrete (C 332) limits the unit weight of aggregates as shown in Table 2.

It should be noted that there is a minimum unit weight for the Group I insulating materials. Experience has shown that materials having unit weights lower than these limits do not have enough strength to permit handling without excessive crushing.

The unit weight of an aggregate is indicative of the average density of the particles and, in turn, is related in a general way to their structural strength. Thus, it is not surprising that normal weight aggregates usually give higher concrete strengths than lightweight aggregates at the same cement content. When lightweight aggregates are considered as a class, however, there is disagreement as to whether differences in the unit weight of such aggregates are significant to the strength of concrete. Kluge, Sparks, and Tuma [*9*] show that the lighter materials usually require more cement in concrete to develop strengths equivalent to that of concrete made with heavier aggregates. On the other hand, Klieger and Hanson [*10*] found no very definite correlation between the unit weight of nine lightweight aggregates and the cement content required for equal compressive strength in concrete. It is probable that such factors as surface texture, shape, and grading can combine to overshadow the importance that unit weight, at least to the extent that this determination reflects the density of an aggregate, may have with regard to the strength of concrete.

Variations in the unit weight of an artificial lightweight aggregate from a single source, however, can be expected to be reflected in the weight and strength of concrete. Such variations can result from differences in degree of burning or other production factors and, thus, usually indicate a change in quality, such as structural strength. For this reason, the uniformity of the unit weight of lightweight aggregates is important to the control of concrete. Each of the ASTM specifications for lightweight aggregates, therefore, provides that successive shipments of aggregate shall have a unit weight that differs no more than ten per cent from that of the original sample submitted for acceptance.

In the case of natural aggregates, lesser importance is normally placed on variations in unit weight of materials from a single source than is customary for lightweight aggregates, even though such variations might be indicative of changes in the character and quality. This is so because unit weight *per se* of normal weight concrete is not usually of concern, and for most aggregates, the strength of concrete containing them is not sufficiently closely related to their structural strength to reflect the changes in this property which might be indicated by unit weight variations. Only to the extent that variations in unit weight may reflect changes in the grading and shape of aggregate, which are sufficient to affect the proportioning of concrete, is uniformity of unit weight of natural aggregates a matter of concern. Moncrieff [*11*], for example, has shown that serious errors in proportioning of concrete designed by weight but batched by volume might arise if changes in bulk density resulting from aggregate grading and shape variations are not taken into account.

DENSITY AND SPECIFIC GRAVITY

In scientific terminology, density is defined as the mass of a unit volume of a material. In applying this concept, the unit volume is considered to be filled with solid material which is free of voids except for those impermeable and permeable voids which are inherent to the particular substance. However, when used in connection with graded aggregates, the term "density" is restricted to mean the proportion of a unit volume that is occupied by aggregate particles as opposed to voids between the particles. Thus, the "maximum density" of a specific graded aggregate is realized when the aggregate particles occupy the highest proportion of space in a unit volume that can be achieved without crushing of the particles or, in other words, when the unit weight of the aggregate is the highest. Maximum density is a function of compactive effort and can be modified by changes in grading, shape, and surface texture.

The density of aggregates is usually expressed either in terms of pounds per cubic foot or as specific gravity, which has the same value as density in the metric system provided the density of water is considered to have the value of unity.

Method of Determining Specific Gravity:

The terms used in referring to density and to specific gravity determinations are defined in the ASTM Definitions of Terms Relating to Density and Specific Gravity of Solids, Liquids, and Gases (E 12 – 61 T). The ASTM Tests for Specific Gravity and Absorption of Fine and Coarse Aggregates (C 128) and (C 127), respectively, are used for determining specific gravity as it relates to concrete aggregates. As applied to aggregates for use in concrete, the specific gravity normally used is the bulk specific gravity on a dry or saturated surface-dry basis. Most of the State highway specifications refer to the saturated surface-dry basis.

A determination of bulk specific gravity requires that the saturated surface-dry weight of the aggregate sample be determined. Uncertainty as to when an aggregate is in this condition, particularly porous or rough textured materials, provides the principal opportunity for variation in results when these methods are used.

Jones and Stephenson [*12*], in discussing the problem of determining the specific gravity of lightweight aggregates, state that absolute specific gravity of expanded materials has no relationship to their volume, since most of the particles have a large number of fully enclosed air voids. They further state that the absorption characteristics of such aggregates are such that the standard test methods do not give consistent results for either apparent or bulk specific gravities. Howard [*13*], in discussing the Jones and Stephenson tests, indicates that a reasonably satisfactory procedure for determining the saturated surface-dry condition of fine lightweight aggregates is to roll the sample in a dry glass jar. When the fines are surface dry, they do not cling to the glass.

The problems associated with determining the absorption of porous materials will be discussed further in a subsequent section.

Because of the difficulty in determining a precise value for bulk specific gravity of lightweight aggregates, the proportioning methods usually used for normal weight concretes are not generally satisfactory for lightweight concrete. To provide a reliable method of proportioning lightweight concrete, Nelson and Frei [*14*] proposed the use of a "specific gravity factor" which can be calculated from any trial batch of concrete and can then be used for calculating volumes in subsequent batches. The specific gravity factor expresses the relationship between the dry weight of aggregate and the space it occupies in concrete, assuming that no water is absorbed during mixing. Since an unknown amount of water is actually absorbed during the mixing process, this factor does not correspond to any of the specific gravities defined by ASTM. Its numerical value should lie somewhere between the apparent and bulk specific gravities. The Recommended Practice for Selecting Proportions for Structural Lightweight Concrete, ACI 613A-59, makes use of the specific gravity factor.

Landgren, Hanson, and Pfeifer [*15*] have pointed out that difficulty may be experienced in controlling the slump and cement content of lightweight concrete when using the ACI 613A design procedure, if the moisture content of the aggregate varies appreciably from that existing in the aggregate at the time the specific gravity factor for the mixture is established. This difficulty is brought about by the fact that the total amount of water absorbed by an aggregate during a prescribed period of time depends to some extent on the amount of moisture initially present in the aggregate. For example, initially damp lightweight particles will usually contain more total moisture than initially dry particles after the same period of water immersion. Thus, if the moisture content of a lightweight aggregate increases on the job, an attempt to control the slump by decreasing the water added at the mixer by an amount equal to the increase in aggregate moisture will usually result in the concrete being too dry.

In the previously mentioned report by Landgren, Hanson, and Pfeifer, it is proposed that the specific gravity factor be based on the moist rather than the dry weight of the aggregate and be determined directly on the aggregates alone by a simple pycnometer procedure. In practice, it would be necessary to determine the relation between the specific gravity factor and the initial moisture content for each source of aggregate. A procedure is presented for calculating the volume displacement of each ingredient of a lightweight concrete mixture at any level of moisture content in the aggregate.

Significance of Specific Gravity:

The concrete technologist regularly makes use of the specific gravity of aggregates in design calculations of concrete mixtures. With the specific gravity of each constituent known, its weight can be converted into solid volume and, hence, a theoretical yield of concrete per unit of cement can be calculated. Some methods of designing concrete make use of the void content of the aggregate as determined by ASTM Test for Voids in Aggregate for Concrete (C 30), which requires determinations of the unit weight and bulk specific gravity.

Proportioning tables for quantities of materials in a batch of concrete are usually based on the use of aggregates having a selected specific gravity. When aggregates are used which have specific gravities varying more than about 0.05 from that on which such tables are based, it is essential that an adjustment be made in the indicated weights to preserve the correct volume relationships.

In the lightweight aggregate field, the specific gravities of the particles in the different size ranges are very important in the design of concrete mixes. Sweet [*16*] was one of the early observers to note, from an extensive investigation of the concrete-making properties of lightweight aggregates, that the finer sizes of lightweight aggregates have much higher specific gravities than the coarser sizes. This has since been confirmed by other investigators. It is evident from this observation that the designer of concrete mixtures can alter the unit weight of lightweight concrete by changing the grading of the fine and coarse aggregates or changing the ratio in which these two fractions are used.

TABLE 3—SPECIFIC GRAVITIES OF MINERALS USED AS HEAVY AGGREGATES.

Mineral	Chemical Composition	Specific Gravity, if pure
Barite........	$BaSO_4$	4.5
Magnetite.....	Fe_3O_4	5.1
Hematite......	Fe_2O_3	5.2
Ilmenite......	$FeTiO_3$	4.7
Goethite......	$HFeO_2$	4.3
Limonite......	$HFeO_2n(H_2O)$	3.8

The advent of nuclear reactors and similar devices has created a need for suitable radiation shielding materials. As shown by Callan [*17*], concrete is an economical material for this purpose. Where space is not a consideration, conventional concrete is generally used, but where space is limited it is essential that the shielding efficiency of the concrete be increased by the use of concrete of maximum density.

This need has led to the study and use of various aggregates having a high specific gravity. Mather and Felts [*18*] have reviewed much of the work done in this field in the U.S. Army Engineer Waterways Experiment Station Technical Report No. 6-635. The natural aggregates usually used for high density concrete are barite and the various types of iron ores. Table 3 shows the specific gravities of these materials when pure. Concretes based on such naturally occurring materials have been produced having densities in the range of 210 to 250 lb/ft^3. The addition of iron or steel fragments to the concrete will, of course, increase the final density of the concrete. Concrete densities in the range of 330 lb/ft^3 can be obtained when the total aggregate consists of iron or steel fragments.

Most specifications for aggregates for concrete limit the amount of such substances as shale, coal, and lignite, because of their deleterious effects. Such low strength particles usually have lower specific gravities than normal aggregates. In areas where chert is present in aggregates, specifications often include a limitation on the amount of lightweight chert. Numerous researches have shown that, for any single source of chert, the soundness of the chert becomes poorer as the specific gravity of the particles becomes lower. Thus, the ASTM Test for Lightweight Pieces in Aggregates (C 123) makes use of the property of specific gravity to separate acceptable from the unacceptable types of particle described above. In this test a heavy liquid having a prescribed specific gravity is used to float off the light deleterious particles which have previously been saturated with water.

Differences in specific gravities of lightweight deleterious material and heavier sound aggregate is also made use of in "beneficiation" processes on a commercial scale. ACI Committee Report 621 mentions three separation methods that make use of differences in specific gravity. One process employs a rapidly moving stream of water which carries away floatable material and allows the heavier material to sink. Only large differences in specific gravity will yield efficient separation. Another process makes use of a jig which is essentially a box with a perforated bottom in which a separating layer is formed by a pulsating water current. Here again, the overlaps in specific gravity range common to both the rejected and accepted fractions can be maintained at a reasonably low figure only if there is a clear distinction between the specific gravities of the objectionable and acceptable particles.

A third process known as heavy media separation involves water suspensions of heavy minerals to separate materials of different specific gravities. The heavy media process requires substantially less difference in specific gravity for efficient operation than is required for the previous methods.

ABSORPTION AND FREE MOISTURE

ASTM Committee C-9 has accepted a definition for inclusion in ASTM Definitions of Terms Relating to Concrete and Concrete Aggregates (C 125), which defines absorption as "the process by which a liquid penetrates and tends to fill permeable pores in a porous solid body; also, the increase in weight of a porous solid body resulting from the penetration of a liquid into the permeable pores." In the case of concrete aggregates, the penetrating liquid is usually water, and the increase in weight is expressed as a percentage of the dry weight of the aggregate and excludes water adhering to an outside surface.

Free moisture is water that adheres to the surfaces of aggregate particles or is held in the voids between particles by the forces of surface tension. Free moisture is expressed as a percentage of either the dry or saturated surface-dry weight of the aggregates.

Method of Testing for Absorption and Free Moisture:

The standard tests for absorption of aggregates are the ASTM Methods C 127 and C 128. In both of these tests, an oven-dry sample is immersed in water for 24 hr and then dried until the free moisture has been removed and only the water held in pores remains, so that the aggregate is in a saturated surface-dry condition.

For coarse aggregate, the method depends on the judgment of the person performing the test to visually determine when a saturated surface-dry condition has been reached. In addition to this source of variation, the fact that large particles dry more quickly than small particles may lead to overdrying of the larger particles, unless the precaution is taken of handling these particles separately and keeping them covered while the remainder of the sample is being dried. These variations are usually small for normal weight aggregates, but they can become significant for those lightweight materials having a rough and porous surface.

Both expanded and granulated slags in particular are of such a cellular nature that a satisfactory saturated surface-dry end point is difficult to establish by the towel drying procedure of Method C 127. A procedure [*19*] based on centrifuging for a definite length of time has been found to provide reasonably reproducible results for slag aggregates. Centrifuging rapidly removes internal water not held by capillary forces. Such water would normally not have time to drain out of the aggregate during towel drying.

The problem of determining a realistic value for the water absorption of coarse lightweight aggregate has been thoroughly studied by Landgren [*20*]. He used a mix absorption test to determine whether previously saturated and surface-dried aggregate particles gain or lose water to a mortar mixture. By this procedure he evaluated the absorption values of a number of lightweight aggregates as obtained by mercury displacement, a kerosene displacement procedure [*21,22*], a rate-of-absorption procedure [*23*], and a modification of ASTM Method C 127. It was found that Method C 127 is suitable for determining the absorption of lightweight coarse aggregate particles provided a selective towel-drying procedure is employed. In the modified procedure, only a few particles are surface dried at a time rather than the entire sample. As particles are brought to a surface-dry state, they are stored in a covered container and are thus not permitted to become further dried by evaporation or excessive toweling. When thus modified ASTM Method C 127 gave values for absorption which agreed fairly well with the values obtained by mercury displacement.

For fine aggregate, the saturated surface-dry condition is defined as that condition which will just permit a cone of aggregate particles to slump. This procedure provides rather reproducible results for natural fine aggregate having a grading suitable for concrete. The end point is more erratic for crushed stone sands because the angularity of the particles does not permit as definite a slump condition as do the rounded surfaces of natural sands. The presence of crusher dust in crushed stone sands also complicates the saturated surface-dry determination because it tends to retain surface moisture longer than the coarser particles which leads to overdrying of the coarser portion of the sample. Although not required by the standard procedure, it is possible to minimize these difficulties considerably by testing individual sizes of the crushed materials separately and then calculating an average absorption by weighting for grading.

Lightweight fine aggregates present even greater difficulty than crushed stone aggregates in regard to determining a saturated surface-dry condition. They not only consist of crushed particles with a high percentage of material passing the No. 100 sieve, but they are more porous as well, which causes the saturated surface-dry end point to be indefinite by any test procedure. As a result, the 0.05 per cent reproducibility specified by ASTM Method C 128 can rarely be obtained on duplicate determinations. Helms and Bowman [*24*] attempted to improve the reproducibility by separating the sample on the No. 100 sieve. The material retained on the No. 100 sieve was tested by the conventional cone technique. A funnel-pour technique was used for the finer fraction. An average weighted absorption was then calculated. In their series of tests they found that absorption determined by ASTM Method C 128 was from 0.7 to 4.3 percentage points lower than absorption determined by the modified procedure using separated fractions.

Free moisture in fine aggregate is determined by ASTM Method C 70. There is presently no standard ASTM method for determining free moisture of coarse aggregates, although the method for fine aggregate contains a note suggesting that it can be used for coarse aggregate with appropriate changes in the size of sample and dimensions of the container. The use of ASTM Method C 70 presumes that the bulk specific gravity of the aggregate is known. As mentioned earlier, the determination of bulk specific gravity is somewhat uncertain for porous materials, such as slag and lightweight aggregates, and therefore the determination of free moisture of these materials is equally uncertain.

Free moisture of both fine and coarse aggregates has long been determined in the field by applying heat to a weighed sample until the sample has either reached a saturated surface-dry condition or a condition of total dryness as indicated by a constant weight. The most convenient approach is to dry the sample until it has a constant weight, in which case the absorption of the aggregate must be known in order to calculate the free moisture. This method is now being considered for adoption as an ASTM method of test. Properly performed, it should measure the moisture content of a sample with a lesser degree of error than is normally associated with sampling aggregate supplies.

Significance of the Absorption and Moisture Tests:

The principal reason for making tests for free moisture and absorption is to determine the amount of water that will be contributed or absorbed by the aggregates in a concrete mixture. The results are used to determine the net total mixing water as a basis for computing the water-cement ratio. Proper control of consistency, yield, and strength of concrete cannot be maintained unless the water in the aggregate is taken into account as well as that added at the mixer. Sometimes the attempt to control total water content is limited to holding the slump constant by changing the amount of water added at the mixer without having a knowledge of changes in the free moisture of the aggregates. However, unless the quantities of aggregates are adjusted to compensate for changes in free moisture, the proportions of fine to coarse aggregate, the quantity and quality of cement paste, and the yield of concrete per unit volume of cement will vary. Such variations will be considerable if the moisture content of the aggregates changes as much as 3 per cent, as it frequently does under field conditions.

The property of absorption has some significance with regard to the soundness of aggregate materials. For a specific rock source, the absorption of the rock will be greater and the specific gravity will be lower for the more weathered and less sound material. A study [*25*] of cherts in Michigan, for example, showed that the 24-hr absorption was the most significant property with respect to predicting freeze-thaw durability. Even when different types of rock are considered, the nondurable types generally have a relatively high absorption, although some durable materials also have a high absorption.

Verbeck and Landgren [*26*] have studied the complexity of factors which determine whether an aggregate can be considered sufficiently durable for use in concrete subject to freeze-thaw cycles. They consider that the durability of concrete depends upon the rate at which the aggregates become critically saturated in the concrete and the nature of the physical response of the aggregate to freezing. Porosity is only one of the many factors which influence these performance characteristics in concrete. Aggregate pore size distribution, size of the aggregate particle, and quality and thickness of the cement paste separating particles from exposed surfaces are also important.

Mather [*27*] has shown that absorption is also statistically related to the results of the magnesium sulfate soundness test when groups of similar aggregates are considered but not necessarily for individual materials. Thus, while the property of absorption undoubtedly has a bearing on the soundness of rocks, it does not appear to be a sufficiently accurate index of this property to be used as a specification item.

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POROSITY

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The pore characteristics of concrete aggregates have been called "... the most important properties of aggregate materials" [*1*].[2] Hardly any property of aggregates, physicochemical or mechanical, is not significantly affected by the pore characteristics. There are many experimental methods for measuring these characteristics or properties related thereto. Frequently the value obtained is strongly dependent on the method, and considerable specificity is needed to avoid ambiguity.

The purpose of this article is to outline these methods and their limitations, especially as applied to aggregates, and to describe the ways in which the pore characteristics affect the important properties of aggregates and of the concrete made therewith.

Because of the generality of the influence of aggregate pore characteristics, an overlap with other papers in this symposium is inevitable.

Definitions

Most naturally occurring solids have an internal porosity. This term refers to the volume inside the individual pieces that is not occupied by solids. It does not refer to the interparticle voids in a packing of the pieces. This interparticle void volume, which is determined by ASTM Test for Voids in Aggregate for Concrete (C 30), is an important property of bulk concrete aggregates but is not discussed in this article.

The total or bulk volume, V_b, of a porous solid is made up of a volume of solids, V_s, and a volume of pores, or voids, V_v. The porosity ϵ is defined as

$$\epsilon = \frac{V_v}{V_b} \quad \text{(1)}$$

and another measure, the voids ratio, R, is

$$R = \frac{V_v}{V_s} \quad \text{(2)}$$

If, as is often true, the void system is partly filled with a liquid and partly with a gas the void volume will be their sum, $V_w + V_a$, to use the subscripts for water and air respectively. The degree of saturation, S_w, is the relative filling of the void volume by the liquid (water)

$$S_w = \frac{V_w}{V_v} \quad \text{(3)}$$

and the absorption, A, is

$$A = \frac{W_w}{W_s} \quad \text{(4)}$$

where W means weight.

Accessory properties needed for an understanding of porous solids are various unit weights. The true or "solids" unit weight, ρ_t, is

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

$$\rho_t = \frac{W_s}{V_s} \qquad (5)$$

There is some ambiguity in terminology here. ρ_t is often called the true density or true specific gravity of the material. Strictly, the density is the mass/volume ratio and specific gravity is the ratio of the mass of unit volume of the material to that of unit volume of water or the weight ratio, if determined at the same gravity value. These distinctions are usually of minor importance for concrete aggregates, since in cgs units they are numerically almost the same because of the nearly unit density of water. In any event, which property is determined will depend on how it is measured, for example, with an equal-arm balance one determines mass rather than weight, and so forth. For simplicity ρ will here be called the "density," while realizing that the term may not be accurately used. These equations are considered further in ASTM Definition of Terms Relating to Density and Specific Gravity of Solids, Liquids, and Gases (E 12 - 61 T).

The bulk density, ρ_b, is

$$\rho_b = \frac{W_s}{V_b} \qquad (6)$$

The bulk-saturated-surface-dry density, ρ_{bssd}, is

$$\rho_{bssd} = \frac{W_s + W_w}{V_b} \qquad (7)$$

and the apparent density, ρ_a, is

$$\rho_a = \frac{W_s}{V_s + V_a} \qquad (8)$$

The last two densities obviously depend on the degree of saturation.

Any consideration of aggregate porosity that goes further than the void volume is soon entangled in questions of pore size. This is an important aspect to many practical considerations of the usefulness of aggregates in concrete. An exact definition is difficult. Most porous solids have a pore structure that is complicated in detail. The voids twist and turn, get larger and smaller, go this way and that, or stop altogether. The pore walls are usually crystal faces of widely varying size and orientation. The "size" of such pores, although a clear enough concept in a rough sense, is difficult to define. Scheidegger [*2*] concluded that the only unambiguous "pore size" was that of the largest sphere that included the point in the pore space in question and lay entirely in that space. While unambiguous, this is not a useful definition, and the dilemma is almost always solved by making a model. An imaginary porous body is constructed that has properties identical to those of the real one (for example, density, porosity, permeability) but that has a pore system of such simplicity that it can be analytically described in detail. By far the commonest model of porous solids is one in which the pore space is made up of a bundle of capillary tubes, perhaps of different sizes. More complicated models have been devised, and the subject is treated in detail in Scheidegger's book [*2*].

Closely related to pore size considerations are the more tractable ones of grain size of the solids. Related thereto is the specific surface, *S*, defined as the surface area of the solids on some unit basis. The three most common are weight of solids, bulk volume, and solids volume, that is,

$$S_g = \frac{A_s}{W_s} \qquad (9)$$

$$S_b = \frac{A_s}{V_b} \qquad (10)$$

and

$$S = \frac{A_s}{V_s} \qquad (11)$$

respectively.

Another way the difficulty of pore size has been bypassed is through the stratagem of the hydraulic radius, which is defined as the cross-sectional area of a pore space divided by its wetted perimeter. It is a term relating to the flow of fluids through a porous solid, but for a pore space saturated with either a gas or a liquid the wetted perimeter becomes the total perimeter, and then the hydraulic radius, m, is

$$m = \frac{\epsilon}{S_b} \qquad (12)$$

If the question of pore size is more or less resolved, it is possible to define a variety or distribution of pore sizes. If a plot is made of the volume of pore space smaller than r, then the slope of this curve, dv/dr, is $f(r)$, the so called pore-size distribution function and is the volume of pores with sizes between r and + dr, or in the limit, the porosity of size r.

If a fluid is flowing through a regular cross section of a porous solid, the permeability, K, of the solid is defined by

$$\frac{V}{At} = \frac{K\Delta P}{\eta L} \qquad (13)$$

where V/t is the volume flow rate of the permeant, A is the approach area of the solid, that is, that normal to the flow direction, $\Delta P/L$ is the hydraulic gradient, in pressure units, causing the flow, and η is the viscosity of the permeant.

A final parameter of interest is the tortuosity, k_t, which is defined as the square of the ratio of the "real" or tortuous length of the flow path of a fluid permeating a porous solid to the bulk length of the flow path, L.

MEASUREMENT METHODS

Porosity:

A distinction is frequently made between the total porosity and that part that is interconnected, which is usually called the effective porosity. For many aggregates a reasonable assumption is that total and effective porosities are equal, but for others, such as many volcanic rocks and some slags, a large part of the porosity consists of isolated cavities, and the effective porosity is less than the total porosity.

In general, any part of the pore space of an aggregate piece that is completely isolated from the exterior is not of concern to questions of durability, since access of water to the pore is usually a necessary precondition for any trouble that may arise. Other properties, such as density and strength, are obviously affected as much by the isolated as by the effective porosity.

Probably the commonest method used in casual investigations of aggregate porosity is to measure the absorption and assume the volume of water absorbed to equal the pore volume, that is, to assume complete saturation.

ASTM Test for Specific Gravity and Absorption of Coarse Aggregate (C 127) is the method for absorption of coarse aggregates and is straightforward except for the question of surface drying. The surface moisture must be dried from the wet aggregate without removing any water from the pores, either by evaporation or by capillary attraction into the cloth used. If the sample has a more or less plane surface, the disappearance of surface moisture is signaled by the loss of the surface sheen or glisten. But for the more usual kinds of samples it is harder to do reproducibly, and this factor is probably the greatest contributor to lack of precision.

For fine aggregate the procedure, ASTM Method of Test for Specific Gravity and Absorption of Fine Aggregate (C 128), arrives at the surface dry condition by allowing evaporation until the particles will no longer cohere after being compacted in a small conical mold.

The absorption value obtained will obviously depend on the length of soaking and other conditions of the experiment. Many methods have been used but only two so frequently that they have become traditional. These are the "24-hr" absorption and the "evacuation and saturation" or "vacuum saturated" absorption. In the former, which is that to which Methods C 127 and C 128 refer, the sample is soaked for 24 hr. In the latter it is dried, evacuated on an aspirator (a better vacuum pump makes hardly any difference in the result), the water admitted, and atmospheric pressure restored. The sample then soaks 24 hr. Naturally, vacuum-saturated values of absorption are larger than 24-hr values, so it is necessary to be specific. If no qualifying adjective is used the 24-hr exposure is assumed.

The main point with respect to the absorption method as a measurement of porosity is that one hardly ever gets complete saturation. Even the relatively drastic evacuation procedure results typically in degrees of saturation of perhaps 90 per cent, and they may be as low as 50 per cent. Only with very open, large-pored aggregates can one assume more or less complete saturation. The volume of absorbed liquid is, therefore, an imperfect measure of an aggregate's porosity.

A common and effective method is to determine the bulk and true densities and calculate the porosity by

$$\epsilon = 1 - \frac{\rho_b}{\rho_t} \quad \text{........} (14)$$

The true density can be determined in various ways. If the aggregate is sufficiently pure chemically, as in a pure limestone, the true density can be obtained from tables or by calculations based on chemical composition. Practically, for aggregates, the material is hardly ever pure enough for this. The usual method, then, is to powder the sample and to determine the density pycnometrically by some procedure, such as ASTM Test for Specific Gravity of Soils (D 854) or Test for Specific Gravity of Hydraulic Cement (C 188).

As in the determination of absorption, the difficulty with these procedures concerns the unknown degree of saturation. Powdering to, say, 100 mesh is usually considered to give a sample that will have complete saturation after evacuation and boiling on the pump at room temperature. The point to the powdering is not so much to destroy void spaces, since most aggregates have pores far smaller than any reasonable sieve opening, but rather to give particles so small that their pores are readily and completely penetrated by the pycnometer fluid under the test conditions. This is probably a reasonable assumption for most aggregate materials, although explicit investigation of the point seems not to have been made.

An alternate way of determining the true density is with a volume change apparatus and a gas as the pycnometric fluid. The volume of the system containing the dry sample is changed by a known amount and the gas pressures are determined before and after the change. Assumption of Boyle's law permits calculation of the volume of solids in the sample. Obviously, any isolated voids will then be counted as solids, that is, the method measures effective porosity, and there is no problem with the degree of its saturation. An advantage is that the method does not require a powdered sample and so is nondestructive. The gas usually used is air, but helium may be better for samples of large specific surface because it is so little adsorbed. An early form of the apparatus was given by Washburn and Bunting [*3*]. Schumb and Rittner [*4*] devised a helium densitometer for use with small samples. A relatively modern apparatus is that of Beeson [*5*]. Commercially available equipment has been designed on this principle.

Bulk density can be determined by dimensional measurement if the sample pieces are regular geometric shapes. Cored cylinders or sawed prisms are convenient. This method is impossible for fine aggregate and frequently impractical for coarse, especially for gravels.

An alternate is to determine the bulk density by the buoyancy method, weighing dry in air and, after absorption is completed, weighing in air and water. This procedure is given in ASTM Methods C 127 and C 128 and again involves uncertainties of surface drying.

Such uncertainties can sometimes be overcome by coating the sample with paraffin to prevent absorption of water, but then a correction for the volume of the coating must be made. Another possibility is displacement of some nonpenetrating liquid, such as mercury, but such procedures are inconvenient, to say the least.

Porosity can be determined visually on a polished or thin section of the aggregate. Since most aggregate materials have a random orientation of their pores, a plane passed through the porous solid will intersect a relative area of void space that is equal to the porosity. Therefore, if the area of pore space in a slice can be determined microscopically the porosity can be determined. The void area can be measured by various camera-lucida or photomicrographic methods. The method has been used on aggregates [*6,7*], concrete [*8*], and building stone [*9*].

Extensions of this idea are the linear traverse and point count methods that are described in ASTM Recommended Practice for Microscopical Determination of Air-Void Content, Specific Surface, and Spacing Factor of the Air-Void System in Hardened Concrete (C 457 – 60 T) for use on the air-void system of concrete. These methods also require a polished or thin section of the material. The linear traverse method is based on the principle that a random line will intersect a fraction of void space equal to the porosity. The point-count method is based on the statistical principle that of a large number of random points a fraction equal to the porosity will fall in the pore space.

These microscopical methods involve problems in the preparation of the section, and they are most tedious and not particularly precise. But the main objection to their use to determine the porosity of aggregates is that one must see the void space before he can count it. At useful magnifications for such sections a void that is smaller than about one micron will not even be seen. Since many aggregates have considerable portions of their pore space smaller than this limit, these methods have been used only to determine the fraction of the porosity larger than some certain size, usually a few microns, that is, the fraction that can conveniently be seen as opposed to that which cannot. For this purpose the method is useful; for the general determination of porosity it is not.

Effective porosity can be determined with the so-called "McLeod gage" porosimeter [*10*]. The method gives a value for the effective void volume in a nondestructive manner by causing the head to be lowered on a dry sample while it is immersed in mercury. The air in the voids expands and leaves the sample and is then measured volumetrically at atmospheric pressure. If the bulk volume of the sample can be obtained, as by the use of a shaped piece for example, then the porosity follows by Eq 1. The method can be combined with the gas pycnometer to give the voids ratio directly on irregularly shaped specimens. This method has been used on aggregates [*11*].

Pore Size:

Determinations of pore size are subject to the difficulties of definition discussed previously. The alternatives seem to be clear. One can either be satisfied with a determination of hydraulic radius or specific surface, which are mutually related by Eq 12, or he can use a model and be satisfied with the results to the extent that the model is realistic.

A separation based on pore size can be made by the various microscopic methods previously discussed. This has been the method used on aggregates to distinguish "macropores" from "micropores" [*6,7*]. For the pores that one can see in such a section, the use of a spherical model will result in a determination of specific surface of the pores. This is the basis for the generally satisfactory use of the linear traverse and point-count methods for the air-bubble parameters of concrete, ASTM Recommended Practice C 457. Here the success is considerable, because the pores in question, the air voids, can be seen, and the model corresponds closely to the fact.

The specific surface of the solids in a porous body can also be obtained from a photomicrograph by a statistical method of Chalkley, Cornfield, and Park [*12*]. A needle of length ℓ is allowed to fall many times at random on the photomicrograph, the magnification of which is n. If the number of times both ends of the needle fall in the pore space is x and the number of times the needle intersects a pore wall is y, then

$$\frac{\ell x}{y} = \frac{4\, V_v}{A_s} \quad \text{.........} (15)$$

and it follows that

$$S_b = \frac{4\, \epsilon y n}{\ell x} \quad \text{..........} (16)$$

All microscopical methods for pore size or specific surface have obviously the same drawback mentioned before: one must be content with what he can see.

One of the most frequently used ways of determining the specific surface of a solid, so effective that it is almost "the" way of doing it, is the sorption method. Much work has been done on sorption in general and on its use to determine the extent of solid surfaces. For a recent review the reader is referred to the book by Young and Crowell [*13*].

The principle is to determine the magnitude of uptake of a vapor by the solid as a function of the vapor pressure and at a constant temperature, that is, the adsorption isotherm. These data are then interpreted by one of several theories, and the result is a value for the surface area of the solids A_s. The theory that has been by far the most useful for obtaining specific surfaces from sorption isotherms is that of Brunauer, Emmett, and Teller (BET) [*14*].

Descriptions of various experimental methods for sorption measurements can be found in many places, for example, [*13,15,32–34*].

The surface measured by this method is obviously only that contacted by the sorbing vapor; only the effective pore space participates, unless the solid is crushed to expose otherwise isolated pores. Further, any pore spaces too small to admit the gas molecules will not be reflected in the determination. This last consideration may be pertinent for the very small gel pores of hardened portland cement paste [*16*], but it is probably not for the generally larger pores of aggregate materials. The usual gases are nitrogen or water vapor. An advantage of the latter is that relatively simple apparatus can be used at room temperatures.

A difficulty with the method for aggregates is caused by their comparatively small specific surface areas. Values typically range from a few hundredths to a few square meters per gram. With such solids it is necessary to use comparatively large samples to minimize experimental error. The use of krypton at −195 C as a sorbate is a useful variant for samples of low-surface area [*17*].

The sorption method has found some use with aggregates and other rocks [*11,18,20,21*].

A second major way of determining the hydraulic radii and specific surfaces of solids is by means of permeability measurements. The topic has been extensively studied. Excellent reviews are books by Carman [*22*] and by Scheidegger [*2*].

The principle involved is that a fluid, either a gas or liquid, in viscous flow through a porous solid is retarded by a drag on the pore walls that is proportional to the magnitude of the solid surfaces contacted. Permeability was defined in Eq 13. When so defined, it is related only to the properties of the porous solid. The quantitative relationship most often used involves a capillary-tube model that has two realistic attributes: the pores have an irregular cross section, and the flowing fluid traverses a path longer than the gross distance between the inlet and outlet faces. When such a model is analyzed using the generalized Poiseuille equation, the result is the Kozeny-Carman equation

$$K = \frac{\epsilon m^2}{k_o k_t} \dots\dots\dots (17)$$

where k_o is a shape factor depending on the cross section of the pores, and everything else has been defined. Equation 17 is equivalent to an alternate form

$$K = \frac{\epsilon^3}{k_o k_t S_b^2} \dots\dots\dots (18)$$

Permeability measurements are relatively simple except for the detail of holding the sample, which is usually a shaped piece of regular cross section, a cylinder, or prism. It must be held so that the permeating fluid traverses it completely and does not leak past the sides. When the sample can be cast in the holder, as with concrete for example, there is no problem, but materials such as aggregates must be cut to the proper form and then sealed in the holder.

Either liquids or gases can be used as the permeating fluid. The use of gases is quicker because of their lower viscosities and is just as good for those substances, such as most rocks, that do not have their structures altered appreciably by drying. It is necessary, however, to make a correction for the enhanced flow or "slip" that takes place when the mean free path of the gas is no longer small compared with the size of pores through which it is flowing [*22,23*].

Once the permeability has been measured the hydraulic radius can be calculated by Eq 17 or the specific surface by Eq 18 if k_o and k_t are known. Early work on packings of spheres showed that the product $k_o k_t$ was about 5. This product is sometimes called the "Kozeny constant." Further analysis showed that the values of its individual members were 2.5 for k_o and 2 for k_t. Carman [*22*] lists values for k_o for various regular cross sections; fortunately it does not vary widely for different shapes. For a wide variety of unconsolidated porous media k_o has been found to be about the same as for a packing of spheres, 2.5 [*24*]. Wyllie and Spangler [*25*] found for consolidated media, such as sandstones and limestones, a value of k_o of about 2.7.

The proper value of k_t, the tortuosity, has been the subject of debate and some experiment. That many porous media might have considerably more tortuous flow cannels than do bead packs was recognized and blamed for failures of the simple (that is, $k_o k_t = 5$) application of the Kozeny equation to consolidated porous media.

Several methods have been proposed for the measurement of k_t. Of these, perhaps the best is the electrical method, the basis of which is the assumption that a flowing fluid and an electric current (when the pores are filled with electrolyte) will follow the same paths. When tortuosities are measured directly, the usefulness of the Kozeny equation is improved, although some k_t values have been recorded that are so large as to be intuitively questionable [*11*,*22*]. The proper value of k_t is the weakest aspect of the Kozeny equation with respect to its use on consolidated porous media, such as concrete aggregates.

In any event the permeability method must be expected to give a relatively low value of the specific surface, because it reflects only the surface in touch with the flowing fluid. Any pore space containing stagnant fluid does not contribute to the viscous retardation. On the other hand, if one is interested in the pore space that supports fluid flow, as for example in studies of the critical size of aggregates in freezing, then the permeability method may give a preferable answer. This illustrates the precept that a test method should be selected critically in order to measure what one is interested in.

The permeability method has been used extensively in the petroleum industry, some on concrete [*26–28*] and handened cement paste [*29*,*30*], but only little on aggregates [*11*,*31*].

Another group of ways to determine the pore size of porous solids can be broadly classified as meniscus phenomena. Across any liquid meniscus there exists a pressure difference, called the capillary pressure, P_c. For a meniscus that is a spherical segment of radius R the capillary pressure is given by

$$P_c = \frac{2\gamma}{R} \quad \text{.........(19)}$$

where γ is the surface tension of the liquid. In terms of the radius r of the tube containing the liquid this equation becomes

$$P_c = \frac{2\gamma \cos \theta}{r} \quad \text{.......(20)}$$

where θ is the contact angle, that is, the angle in the liquid between the liquid and solid surfaces. The basic idea of the various meniscus methods for pore size is to measure P_c, or some property associated with it, and apply Eq 20 to obtain r. It is, of course, necessary to know γ and θ. The surface tension is no problem, except that the change that may take place in very small capillaries is unresolved [*35*]. Practically, what is done is to assume the value of surface tension of bulk liquid and, except for liquids like mercury for which it is most obviously untrue, that $\theta = 0$. Unfortunately, the assumption of zero contact angle is more often wrong than right, at least for water, which is the most important liquid in experiments on aggregates. High-energy surfaces, like the minerals that make up aggregates, almost always have adsorbed films of oil or what not from the air that give finite contact angles with water. This is a drawback to the method as an absolute but not necessarily as a relative measure of pore size.

Since each pore of a different size will have a different capillary pressure across a liquid meniscus it contains, these methods are used for the determination of pore-size distributions of porous solids. All that is needed is to determine the capillary pressure, and thereby the pore size, as a function of the degree of saturation; one can then construct the aforementioned cumulative volume versus pore size curve and, from it, the pore size distribution curve.

The dependence of the results on the model should be remembered. For example, both a multisize capillary tube model and one with identical pores of conical shape could give the same pore size distribution curve and the same experimental result. Here again the usual procedure is to use the capillary-tube model.

Many experimental methods have been used. They involve bringing about a change in the amount of one phase in the pore system by forcing in a displacing phase. A wetting phase is retained in the pores by a pressure equal to the capillary pressure. A nonwetting phase ($\theta > 90$ deg) is denied entrance to the pore by the same pressure.

If the wetting phase is water, the nonwetting phase is usually air. The air can be forced in to displace the water, or the pressure difference can be established by putting a tension or "suction" on the water, whence comes one of the names of this sort of experiment. The displaced water is then measured volumetrically at various pressure differences, and these provide the data for the so-called capillary curve, that is, the cumulative volume versus the pore size. Rose and Bruce [*36*] reviewed the technique and showed a general form of the apparatus. There have been many variations. This technique has been much used in other fields [*36–38*]. Essentially this was the method used on aggregate materials by Blanks [*39*], and his results confirmed the microscopical results showing the influence of micropores on frost durability.

A second major technique is to use nothing, that is, a vacuum, as the initially saturating phase and to use mercury as the displacing phase. This technique forms the basis of the so-called mercury porosimeter, which has become almost a standard method for these purposes. In this equipment the mercury is forced into the evacuated sample, and the amount that goes into the pores is measured as a function of the pressure causing ingress, that is, the capillary pressure. The pressure is usually applied hydraulically after accidents with gas-activated equipment. The idea was advanced long ago by Washburn [*40*] and was developed by Ritter and Drake [*41*].

Examples of the use of the mercury intrusion method on rocks are the work of Purcell [*21,42*]. The method has been applied to concrete aggregates by Hiltrop and Lemish [*43*]. Several commercial instruments using this principle are available.

Another method that depends on capillary pressure combines the meniscus effects with permeability. This is a dynamic method in which a liquid, usually water, is allowed to enter the pore system of the dry solid by capillarity. The progress of the liquid is retarded by viscous forces, so the forces causing both the advancement and the retardation are dependent on the pore size.

The quantitative measure of this effect was called by Powers and Brownyard [*34*] the absorptivity, K_a. If the approach area normal to the capillary ingress is A and the volume of permeant absorbed in time t is V then K_a is defined by

$$\left(\frac{V}{A}\right)^2 = K_a t \quad \text{........(21)}$$

The absorptivity can be related to the pore size or the specific surface of a model of the porous solid. The method has been used on hardened cement paste [*34*] and on limestone aggregates [*11*]. It requires only a balance and a stopwatch and is easy to perform.

One of the effects of the capillary pressure across the meniscus of a wetting liquid in a pore is a reduction in the vapor pressure below that of bulk liquid. This means that liquid can exist in equilibrium in small pores at "humidities" less than unity. Further, whether or not liquid will be stable in a pore at a given humidity is a function of the size of the pore. The quantitative expression of this so-called capillary condensation effect is the Kelvin equation

$$\ln\left(\frac{P}{P_o}\right) = \exp\left(-\frac{2\gamma \cos\theta\, M}{r\, \rho\, RT}\right) \ldots (22)$$

where p/p_o is the relative vapor pressure or humidity, over liquid of surface tension γ, density ρ, molecular weight M lying at contact angle θ in a pore of radius r at temperature T.

This effect is used to interpret the relatively high-pressure portion of the adsorption isotherm. The relationship between the volume of liquid in the pore system and the corresponding pore size, that is, the pore size distribution curve, can then be derived from these data.

The use of the Kelvin equation has been discussed in various reviews [*35,44*] and modified in appropriate ways [*45,46*].

The main difficulty with the use of this method on concrete aggregates is, in addition to problems of surface tension and contact angle, that the pore sizes that can conveniently be investigated are small. Equation 22 shows that the equilibrium vapor pressure over large pores is almost p_o. This high-humidity region of the isotherm is difficult experimentally, so usually the investigation must be confined to comparatively small pores. In this respect mercury intrusion is preferable, because the larger pores can be investigated at lower pressures. A convenient possibility might be to use both methods, each to its best advantage, that is, the mercury porosimeter for the larger pores and capillary condensation methods for the smaller ones.

Other methods for specific surface and pore size exist. Small angle X-ray scattering, heat of immersion, rate of dissolution, and radioactive and electrical methods can be mentioned. But these have been so little used on the materials of interest here that they will not be discussed.

A final small word of caution might be appropriate after this discussion of methods for determining pore sizes or specific surfaces of porous solids. With respect to concrete aggregates, most of the properties of consequence are not so much related to these parameters directly as to the properties that they control. The permeability, capillary attraction and retention, and so on are more significant to questions of frost durability of aggregates than is the pore size itself. So it may be pointless to calculate a pore size or size distribution from permeability or meniscus experiments, involving as they do the choice of a usually most imperfect model.

SIGNIFICANCE OF POROSITY OF AGGREGATES

The purpose of this section is to indicate the reasons for the importance of the porosity of aggregates. Many properties of the aggregate and, therefore, of the concrete are importantly or even critically affected by its porosity. Most of these significant aspects have been reviewed before, and it is not the intention here needlessly to repeat. The aim is, rather, to put things in perspective and treat material that has not before been summarized. This last is chiefly the generalized theory of freezing and thawing of aggregates in concrete, which has emerged into a much clearer light than it enjoyed ten years ago when much of this material was considered in the paper of which this is the successor

[*19*]. The reader is referred to this earlier paper for other references.

These matters have also recently been discussed in a review and annotated bibliography by Larson et al [*48*].

Broadly speaking, the porosity of a concrete aggregate affects the behavior of the concrete for one or both of two reasons—either because the pore space decreases the volume of solids in bulk volume of the aggregate or, more importantly, because the pore space permits the ingress and retention of water or aggressive solutions.

The effects of the first of these two classes are mostly obvious. The influence on density is of importance only indirectly in such matters as mix proportioning and some special types, most notable of which is lightweight-aggregate concrete.

Other things being equal the strength and, consequently, the abrasion resistance of the aggregate will be smaller the greater its porosity. This influence is probably not simply one of a reduced cross section to bear the load. Abrasion resistance is an important property of aggregates that is related to strength and porosity. The Los Angeles abrasion test, ASTM Test for Resistance to Abrasion of Small Size Coarse Aggregate by Use of the Los Angeles Machine and Test for Resistance to Abrasion of Large Size Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 64 T and C 535, respectively), is part of most aggregate specifications, for example ASTM Specification for Concrete Aggregates (C 33). This property is recognized to be of major importance in determining the degree of breakdown an aggregate can be expected to undergo during mixing of concrete or compaction of base course, ballast, and so on.

Except for those that are grossly weak, the strength of the aggregate is not considered to be a primary factor governing the strength of concrete. The strength of the cement paste, which is intimately related to its own porosity [*49*], and the strength of the paste aggregate bond are more important [*50*]. Little has been done on this last question, but it is logical to assume that the roughness and the porosity of the surface of the aggregate play an influential part in the development of the interfacial bond with the paste [*51*].

The porosity of aggregates also affects their other mechanical properties, such as modulus of elasticity and Poisson ratio. These properties in turn are important to the same properties of the concrete, which are additive functions of the properties of its constituents [*52*].

The mechanical properties are influential in the behavior of concrete in shrinkage. The theory of Pickett [*53*] predicts the influence of the elastic modulus and Poisson ratio of the aggregate in determining the degree of restraint of the aggregate on the shrinking paste. Explicit tests of the effects of porosity seem to be lacking, as is the situation with many other properties of concrete that are affected by the porosity of the aggregate. A principal reason for this lack is the difficulty of obtaining aggregate samples that are identical except for differing porosities. Until now, investigators have mostly used what they could get, and although different aggregates had different porosities they had other properties different too.

Of even greater significance is the second of the two general categories, the influence of the pore system on the acquisition and retention of water or solutions by the aggregate piece and the control of the movement of these liquids through the material. Chief among such effects is the great influence the aggregate pore system had on the frost durability of the concrete.

Porosity is important to questions of

proportioning concrete because of its control over the absorption of the aggregate. Any mixing water that is abstracted by the aggregate during mixing and placement must be considered in the mix proportions. Usually this is done in an effective, if approximate, fashion by the application of relatively small correction factors. But in the proportioning of lightweight aggregate concrete the needed correction factor frequently becomes large and indeterminate because of the large absorption that can occur with lightweight aggregate. A realistic absorption test for lightweight aggregate has recently been considered by Landgren [*54*].

When the pores of an aggregate are wholly or partially saturated with water, certain properties can be considerably changed. Because of the relatively large differences in thermal properties between water and dry aggregates, the values of heat capacity, thermal conductivity, thermal diffusivity, and expansion coefficient of an aggregate change considerably with the absorption of relatively small amounts of water. See [*19*] for some references to earlier work. Since the thermal properties of aggregate have yet to be shown to be especially important to concrete durability, these effects of porosity are probably minor.

In addition to the previously mentioned influence of the mechanical properties of the aggregate on the shrinkage of concrete, a few aggregates undergo excessive volume change themselves on wetting and drying and, therefore, can contribute greatly to the shrinkage, basically because by shrinking the aggregate fails to restrain the shrinking of the paste [*55*]. Little work has been done on these aggregates, and, except possibly for the influence of clay minerals, few generalizations can be stated concerning the types of materials that may be particularly subject to this difficulty. However, it is logical to assume, in line with other systems showing large wetting-drying volume change, that the presence of a high surface area, finely-pored material is involved.

The alkali-aggregate reaction requires contact between the aqueous phase in the concrete and susceptible minerals in the aggregate. Presumably a greater internal surface area of the aggregate would mean an enhanced degree of reaction. But greater reaction does not necessarily mean greater difficulty and lack of durability. The Powers-Steinour hypothesis [*56*] postulates that an extensive reaction coupled with available lime nearby in the solution will result in the formation of a safe, nonswelling gel, as occurs with pozzolans or greater-than-pessimum proportions of reactive aggregates. With respect to the interior of aggregate pieces, however, the distance of the lime is so great that its potential protection would probably not develop. Accordingly a high internal surface area of the aggregate probably would result in greater difficulty. There are hardly any data on which to base a conclusion.

Foremost in the list of significant effects that the porosity of aggregates has on the properties of concrete is freezing and thawing durability.

That the damaging effects of freezing of building materials depend on the access of water and on the acquiring of a high degree of saturation has, of course, been obvious all along. But the details of the process and the quantitative evaluation of the various mechanisms have been by no means clear, and only recently has the matter been reasonably well-understood.

Study of frost durability of aggregates in concrete has been given impetus by the growing scarcity of "good" aggregates and the necessity to use materials that would have been bypassed in former days. Part of this research effort was to develop better test methods to predict the quality of aggregates.

Early work on the subject of freezing and thawing durability of concrete and aggregates has been summarized in the previously cited reviews [*19,48*] and in many others and will not be repeated in detail here.

The earliest work was on building stones, bricks, and ceramic tiles. The work in this field has been recently reviewed by Butterworth [*58*]. The volume change on freezing of water implies that a degree of saturation larger than about 0.91 should be harmful. Some work has shown [*57*] lower saturations to be dangerous because of nonuniform distribution of water in porous bodies with a variety of pore sizes. A liquid will remain preferentially in the smaller pores because of their larger capillary potential.

This so-called degree of saturation or milk-bottle-in-the-winter hypothesis was, of course, the earliest and obvious one for destructive frost action. If the water cannot get out and the degree of saturation is too high, disruption of the material must occur, because the pressures developed on freezing will be far larger than any such material can stand.

And, although this mechanism has been greatly amplified by subsequent work, it still stands in a somewhat negative sense. If the degree of saturation of an aggregate particle is less than somewhere in the region of 90 per cent, there will be no damage to the concrete from this source. It should be remembered that, as saturation is ordinarily determined, an inhomogeneous material, such as most gravels, may have some individual pieces critically saturated although the value determined on the over-all sample may be considerably lower.

The degree of saturation hypothesis is the basis for a widespread dependence on absorption tests to indicate an aggregate's probable performance. While the absorption value is not, of course, as precise an indicator as is the saturation, for most aggregates of the usual compositions and porosities the absorption is good enough, especially considering the simplicity of the test method, to be a fairly useful indication of the likelihood of trouble. Indeed there is some question of how much the subsequent work on test methods has improved upon it.

Early work on the degree of saturation of aggregates substantiated the findings in other fields. Work on limestones [*59*] showed that almost all with a vacuum saturation of over 90 per cent had poor durability histories on concrete pavements, while the converse was true for those with lower saturations. These findings have been substantiated by other studies, for example, [*60,61*].

In this connection it should be pointed out, however, that for any porous body there is a porosity value small enough so that even if the pores are completely saturated and the water freezes the volume change will be accommodated by expansion of the solid. Verbeck and Landgren [*62*], in what is probably the most important recent analysis of the whole question of frost resistance of aggregates, gave the name of elastic accommodation to this effect. A relatively simple calculation shows the critical values for aggregates of ordinary strengths and elastic properties to be absorptions of a few tenths of a per cent and, therefore, porosities of up to perhaps half a per cent. These properties are met occasionally, by some quartzites for example, and such rocks should be immune from frost damage under any conditions.

The degree of saturation hypothesis has, however, limitations. A necessary condition is that the water has no escape from the region in question. This is a condition hardly ever met in concrete. There is almost always somewhere for the excess water, that corresponding to saturations greater than 0.9, to go. Both the external boundaries of the concrete and any internal voids that are empty are such "escape places."

Considerations of this sort with respect to the freezing of hardened portland cement paste led Powers [*63–65*] to his hydraulic pressure hypothesis of frost action. This hypothesis was a major advance in understanding these mechanisms and is no less true for the aggregate component of concrete than for the paste. The only difference is quantitative.

The essence of the hydraulic pressure theory is that water forced out of a critically saturated region by the expansion due to freezing will move through a permeable porous solid. If the combination of speed of water movement, which is related to the speed of freezing, and distance the water must travel to an "escape place" is too great, the hydraulic pressure generated by this movement will be large enough to fracture the matrix. The principal function of entrained air in concrete is to give a paste that has voids spaced closely enough so that this critical distance is not exceeded [*66*].

The critical distance for hardened paste under normal conditions of freezing is roughly 0.01 in. This distance is proportional to the permeability of the porous medium in question. The main reason the value is so small for hardened paste is its extremely low permeability [*29*].

The permeability of most rock used as aggregate is much greater than that of hardened paste. But the critical distance of some is small enough to be of the order of a maximum size of coarse aggregate usually used in concrete. These rocks are the typical popout materials, lightweight cherts and shales, and others that give such difficulty close to the surfaces of exterior concrete. Verbeck and Landgren [*62*] calculated a critical distance of $\frac{1}{2}$ in. for a typical chert. If the aggregate particle is larger than this (and critically saturated), it should fracture internally on freezing in a "critical size" type of failure.

This is just the sort of behavior that is observed with popouts. Typically a flake of mortar comes out and retains about half the aggregate piece. This behavior shows that the rock fractured internally just as the hydraulic pressure mechanism predicts. Furthermore, if this mechanism is valid there should be more trouble with a larger coarse aggregate than with a smaller one that may be smaller than the critical distance. Again, field experience confirms the expectations; larger aggregate pieces give much more difficulty from popouts than do smaller ones.

The critical size theory is well-established, as much for aggregate as for paste, and seems to explain certain phenomena. But there are others that it fails to explain. Many aggregates that are nondurable in frost exposure have permeabilities so large that their critical sizes are far larger than any piece used in concrete. The example of Verbeck and Landgren [*62*] is a dolomite that would have a critical size of 33 in. No internal damage to such an aggregate can occur by the hydraulic pressure mechanism. Yet many aggregates with such properties do cause trouble in concrete.

If a piece of aggregate is critically saturated and a freezing front passes across it, the excess water must be expelled. If, in the course of this progress, the critical distance is exceeded, the piece will fracture internally. But even if the aggregate is not so affected, the excess water will be expelled into a paste of very low permeability and small critical distance.

This is the mechanism that was invoked by Verbeck and Landgren [*62*] to account for nondurability of large-critical-size aggregates. Analysis of this expulsion-into-the-paste situation shows that a completely nonair entrained paste should fail disastrously from this cause, because if the aggregate is critically saturated the paste is sure to be so also. Any voids in the paste will mitigate the trouble to some extent. According to this mechanism, then, air-entrained concrete should be more free of this type of difficulty than nonair-entrained concrete. There is some evidence [*47*] that entrained air does help in such a situation. But it is usually considered that entrained air cannot be depended on adequately to correct a nondurable aggregate and result in sound concrete.

This mechanism also implies that, as with the critical-size failure, a larger piece of aggregate will be worse, because it will contain more water to be expelled into the paste. One can calculate a certain size, and aggregate smaller than this should be safe from this kind of difficulty. The value depends on the porosity and degree of saturation of the aggregate and the air content, capillary porosity, and critical distance in the paste. Again, the size that separates the harmful from the harmless turns out to be roughly a maximum size of coarse aggregate commonly used in concrete.

Since critical distance and pore size are both associated with permeability, they must also be related to each other. Early work on building stones by Schaffer [*9*] led to the hypothesis that those with a relatively large amount of porosity smaller than 5 μ in "size" were nondurable. How the particular value was selected is uncertain, and it may have been determined pragmatically because of the microscopical method that was used. This value was, however, somewhat uncritically applied to concrete aggregates and used to separate "macropores," which are supposedly all right, from "micropores," which are supposedly not.

Work with a capillary pressure apparatus [*39*] seemed to confirm this view. Further status was lent to it by Sweet [*6*], who found the voids ratio of pores smaller than 5 μ to be a critically separating parameter of aggregates with good and with bad field histories. Work with the mercury porosimeter by Hiltrop and Lemish [*43*] showed again that aggregates with poor field histories had a preponderance of small pores, but the size region that was diagnostic was smaller than that derived from the microscopical studies by perhaps a ten factor.

Much of the work concerning pore sizes was done before the distinctions between the critical-size and the expulsion-into-the-paste mechanisms were clear. With those rocks subject to a critical size failure the permeability and hence the pore size is a factor of highest importance, because it determines whether the size of the aggregates exceeds the critical distance or not.

With the kind of aggregates subject to the expulsion-into-the-paste difficulty the pore size is of no importance once the piece of rock has become critically saturated. At this point all that matters is whether the paste voids can accommodate the excess water within a critical distance in the paste. So with this type of material the crucial factors are those that determine whether or not the rock will become critically saturated. Naturally, these same factors are important to the critical size or any other type of failure, because for any of them to occur the rock must be critically saturated.

Therefore, while the type of failure one gets depends on the permeability, whether or not one gets any failure will depend on the several factors that determine the degree of saturation of the aggregate.

If the rock is mixed into the concrete while in a quarry-wet or stream-wet condition the probability of high saturation and, therefore, of some kind of failure in freezing is high. On the other hand, if the rock has dried out then the amount of water it absorbs will depend on its own properties and on those of the mortar that surrounds it.

Considerations of this sort have led Powers [*65*] to propose a test method for frost durability of concrete that is essentially an *in situ* absorption test that measures the length of time for whatever parts of the concrete that may become critically saturated to do so. The method has been used in a few instances [*67*].

The more impermeable the mortar surrounding the aggregate pieces, the better. The principal factor is the water/cement ratio [*29*]. This is, in fact, a major reason for using a low water/cement ratio. Also, any cracks in the mortar will obviously provide a ready entrance of water to the underlying pieces of aggregate. All those aspects of good practice in mix proportioning, finishing, curing, and structural design that make for crack-free concrete are, therefore, important in this regard as well as in others.

The property of the aggregate piece itself that determines its saturation in the concrete should be its absorptivity rather than its permeability. Pore size works in both directions; the smaller the pore size the greater the capillary potential but the greater also the viscous drag denying absorption. The former factor operates by the first power while the latter is proportional to the square of the pore size. Therefore, a rock with larger pores will have a higher absorptivity than one with smaller. It will also have a higher permeability and a lower capillary potential. This means that a rock with smaller pores will acquire water more slowly but will retain it longer and more tenaciously than will a rock with larger pores. Just which effects predominate will depend on the stage of the process that is most critical with respect to the freezing.

The interrelationship of the aggregate with the other components of the concrete in determining the frost durability is the reason for the difficulty in finding a test method that can be used on the aggregate alone.

Many have been tried with limited success. In addition to those studies already mentioned, a little work [*11*] has shown that aggregates with poor field history had higher absorptivities, by orders of magnitude, than did those with good performance. These samples were limestones with critical sizes so large they all fell within the expulsion-into-the-paste type.

In this same connection, it should be pointed out that the two different mechanisms, critical size and expulsion, are reflected in the results of unconfined freezing and thawing tests and in their lack of general applicability. A critical-size failure will occur whether the aggregate is in concrete or not. These materials will show a large loss in unconfined freezing and thawing, while the rocks subject to the expulsion difficulty will not.

Mention should be made of a final mechanism. Powers and Helmuth [*68*] adapted to cement paste an older theory of frost action in soils that leads to lens formation and frost heave [*69*]. This theory has been amplified by others for soils, for example, [*70*], and has recently been considered in a general thermodynamic treatment for all porous solids [*71*].

The principle is that unfrozen water will be attracted to ice at a subfreezing temperature and will generate distending pressures that will cause lenses and heave in soils and cracking in concrete. In the case of cement paste the ice is in the capillary pores, and the unfrozen water is in the gel pores. Powers and Helmuth [*68*] called this the gel water diffusion mechanism. It does not depend on a volume increase on freezing and works as well for normal liquids that contract on freezing as it does for water.

There is no reason why this mechanism should not be operative for ice in aggregate pores at the paste-aggregate interface. Presumably gel water could diffuse to such bodies of ice as well as to any other. No work has been done on this aspect.

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TOUGHNESS, HARDNESS, ABRASION, STRENGTH, AND ELASTIC PROPERTIES

By D. O. Woolf[1]

The mechanical properties of aggregates may or may not have an effect on the strength or durability of concrete. Since concrete is an assemblage of individual pieces of aggregate bound together by a cementing medium, its properties are based primarily on the quality of the cement paste. Its strength is dependent also on the bond between the cement paste and the aggregate. If either the strength of the paste or the bond between the paste and the aggregate is low, a concrete of poor quality will be obtained irrespective of the quality of the aggregate. But when cement paste of good quality is provided, and its bond with the aggregate is satisfactory, then the mechanical properties of the aggregate will influence the strength of the concrete.

The tests considered in this paper are applied ordinarily to coarse aggregate or to rock from which aggregate is produced. Some of these tests, particularly the toughness and hardness tests, may not be mentioned in present-day specifications for the quality of rock. However, the hardness test does appear suitable for determinations of the rate of wear of concrete prepared with various types of aggregate. Of special consideration may be concretes prepared with natural and lightweight aggregates. Studies recently made in the laboratories of the Bureau of Public Roads have shown that the Deval abrasion test is more suitable than the Los Angeles abrasion test for determining the quality of lightweight aggregates. And, of course, the Deval abrasion test is still used by some states to determine the quality of natural aggregates. Little immediate use can be seen for the toughness test, but it is described for the benefit of those who read of it in old publications or who may find requirements for toughness in present-day specifications. The present studies of increasing the resistance of concrete to impact by the addition of fine steel wires or nylon fibers to the concrete may cause an increase in interest of a toughness test for concrete, possibly based on the design of the test for rock. The method of test for toughness of rock certainly should not be dismissed as archaic.

Toughness

With respect to concrete aggregates, toughness is usually considered the resistance of the material to failure by impact. The test as described in ASTM Test for Toughness of Rock (D 3)[2] is made on small cylinders drilled from ledge rock or large pieces of aggregate. The test values are empirical and indi-

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[2] Now recommended to be deleted due to lack of use or lack of reference in ASTM standard specifications.

cate the distance in centimeters through which a 2-kg hammer falls to cause failure of the specimen.

At one time when horse-drawn or steel-tired traffic was common, determinations of the toughness of rock were considered of importance. With the virtual disappearance of this traffic, the need of a direct test of resistance to impact became remote. The method, however, has been found useful for tests on specimens too small for testing by other means, or for the classification of aggregates with respect to weathering. With careful operation of the test procedure, the results can be used to show whether the material in question is fresh or has been affected adversely by weathering and solution. Average values for toughness of the more common types of rock used as concrete aggregates are given in Table 1.

Attention is directed to the use of petrographic methods for the determination of the extent of weathering of rock. However, as indicated in ASTM Recommended Practice for Petrographic Examination of Aggregates for Concrete (C 295), extreme care must be exercised in the selection and preparation of the slide examined. The chances are quite great that the small section of rock prepared for petrographic examination will represent only the better portion of the rock. More indicative results may be found in the toughness test.

TABLE 1—AVERAGE VALUES FOR PHYSICAL PROPERTIES OF ROCK.[a]

Kind of Rock	Toughness		Hardness		Loss by Abrasion			
					Deval Test		Los Angeles Test	
	Number of Tests	Avg	Number of Tests	Avg	Number of Tests	Avg, per cent	Number of Tests	Avg, per cent
Amphibolite	70	14	56	16	87	3.9	30	35
Basalt	203	19	192	17	203	3.1	24	14
Chert	29	12	29	19	78	8.5	6	26
Diabase	285	20	253	18	340	2.6	63	18
Diorite	48	15	45	18	60	3.1	...	...
Dolomite	612	9	586	14	708	5.5	134	25
Felsite[b]	127	17	118	18	150	3.8	9	18
Gabbro	42	14	38	18	45	3.0	4	18
Gneiss	386	9	365	18	602	5.9	293	45
Granite[c]	703	9	589	18	718	4.3	174	38
Limestone	1315	8	1209	14	1677	5.7	350	26
Marble	188	6	162	13	175	6.3	41	47
Quartzite	161	16	146	19	233	3.3	119	28
Sandstone	681	11	613	15	699	7.0	95	38
Schist	212	12	180	17	314	5.5	136	38
Syenite	32	14	26	18	31	4.1	14	24

[a] From "Results of Physical Tests of Road-Building Aggregate" [*9*].
[b] Including andecite, dacite, rhyolite, and trachyte.
[c] Including granodiorite, pegmatite, and unakite.

Because the toughness test is made only on shaped cylinders that are drilled from the material under test, the test can not be made on aggregate of the sizes usually used in concrete. Several attempts to develop a method of test for sized aggregate have been made. Possibly the most successful of these resulted in a readily portable apparatus known as the Jackson nutcracker in which single pieces of aggregate are subjected to impact by a freely falling steel ball [*1*].[3] A

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[3] The italic numbers in brackets refer to the list of references appended to this paper.

number of refinements of the apparatus have been made to simplify the test procedure and to permit testing material as small as $\frac{1}{2}$ or $\frac{3}{8}$ in. The apparatus was used in an investigation of soft pieces in aggregate [*2*]. It was concluded that, for complete information, three different sizes of steel balls were needed to test aggregate ranging in size from 2 or $2\frac{1}{2}$ to $\frac{3}{8}$ in. With some changes that have been contemplated, the method is considered well adapted for field or laboratory use in determining the quality of aggregates as measured by their resistance to impact, and with suitable allowance for materials which are hard but brittle, the method would also be useful in the determination of soft pieces of aggregate.

HARDNESS

As usually considered in connection with aggregates for concrete, hardness refers to the resistance of the material to surface abrasion. No method for hardness tests on aggregates has been adopted by ASTM. Many state highway laboratories have used a Dorry hardness machine for tests of the hardness of rock. In this test, a cylindrical core of rock is subjected to surface wear by finely crushed quartz carried by a revolving metal table [*3*]. The loss in weight of the rock after 1000 revolutions of the table is determined and the hardness expressed as an empirical value:

$$\text{Hardness} = 20 - \frac{\text{Loss in grams}}{3}$$

No particular basis for this formula is known. It is possible that it was developed to have the test value agree closely with that for the "French coefficient of wear" obtained in the Deval abrasion test.

Like those for the toughness test, the values obtained in the hardness test were considered of significance when steel-tired traffic was common. With disappearance of this traffic, the hardness test was discontinued, and no mention of it is found in the methods used by the American Association of State Highway Officials as early as 1928. Hardness tests on rock were continued by the Bureau of Public Roads, however, until 1934.

In themselves, hardness test values are of interest for comparison, but they do give information concerning the effect of weathering and alteration for particular classes of rock, and as mentioned earlier, the procedure can be used to indicate the resistance of concrete to surface abrasion. Average values for the principal kinds of rock used as concrete aggregates are shown in Table 1. Materials giving Dorry hardness values appreciably below those shown may be considered of questionable quality.

Mention should be made of the use of the scleroscope for measuring the hardness of nonmetallic materials. Extensive use of the Shore scleroscope has been made by the Bureau of Mines [*4*] in a study of the physical properties of mine rock, and considerable data of scleroscope hardness of rock are given by Wuerker [*5*]. In the use of this instrument, the hardness of the material is measured by the rebound of a diamond-tipped hammer dropped vertically on the test surface. Since the diamond point has a diameter of 0.03 in., it may strike a single mineral crystal in the rock. It is necessary, therefore, to make a considerable number of tests at random locations on the specimen to obtain a good value for the hardness of the material. The method appears to be suitable for use on aggregate, even material of a heterogeneous nature, since with judicious sampling the fragments tested may be held to a reasonable number.

And due to the small size of the apparatus, the method may be found quite suitable for use in the field.

ABRASION

Deval Abrasion:

The Deval abrasion test has been used since 1878 as a measure of the quality of rock. In 1908 it was adopted by ASTM as Test for Abrasion of Rock by Use of the Deval Machine (D 2) and has been revised but slightly since then. A method of test for gravel was adopted in 1928 as ASTM Test for Abrasion of of Graded Coarse Aggregate by the Use of the Deval Machine (D 289); this has been revised several times to permit the testing of crushed or uncrushed gravel as well as crushed stone and crushed slag. A tabulation of requirements for coarse aggregate for concrete shows that in 1958 six state highway departments used the Deval test, or a modification thereof, for the acceptance of materials and, in some cases, in addition to the Los Angeles abrasion test [*6*].

In the Deval test for rock (ASTM Method D 2), approximately cubical pieces of rock with sharp edges and corners are tumbled in a cast iron cylinder for 10,000 revolutions. The sample is then sieved on a No. 12 sieve with openings about $\frac{1}{16}$ in. square, and the weight of the material passing the sieve is expressed as a percentage of the original weight of the sample. A "French coefficient of wear" may also be calculated by dividing 40 by the percentage of wear.

In ASTM Method D 289 for gravel or crushed aggregate, a graded sample is used with an abrasive charge of steel spheres. Five different gradings are now permitted with maximum sizes of 2, $1\frac{1}{2}$, 1, $\frac{3}{4}$, and $\frac{1}{2}$ in.

As seen in Table 1, the Deval percentages of wear for many of the commonly used types of rock, such as, limestone, dolomite, marble, gneiss, schist, and sandstone, do not differ very much. Because of this, the Deval test does not provide a basis for predicting the preformance of rock or rock types as aggregates for concrete. The loss in the Deval test can be used for comparative purposes as an approximate measure of the quality of the rock. Many specifications in the past permitted a percentage of wear of 8.0, or a French coefficient of 5.0, as an indication of rock of suitable quality for use in concrete.

The Deval test for graded coarse aggregate appears to be more severe than that for rock and normally gives a higher percentage of wear for material of presumably identical quality. It has been customary to consider rock with a loss of about 7 per cent as determined by ASTM Method D 2 to be of the same quality as gravel with a loss of about 15 per cent as determined by ASTM Method D 289. This relation is not supported by experimental data. In separate investigations made by two laboratories, rock that had been milled to the shape of gravel and tested by Method D 289 had only slightly more wear than rock from the same quarry tested by Method D 2. It must be concluded that the results of the two methods are not directly comparable. The difference in the shape of the pieces tested, the greater amount of impact in Method D 289, and the usual presence of quartz in gravels cause the loss for the graded aggregate method to be appreciably greater than that for Method D 2.

A principal objection to the use of Method D 289 for the acceptance of materials concerns the different losses that might be obtained for the same material. The method permits the use of

TABLE 2—EFFECT OF GRADING OF TEST SAMPLE ON LOSS IN DEVAL ABRASION TEST METHOD D 289.

Size of Particle	Grading A	Grading B	Grading C	Grading D
	COMPOSITION OF TEST SAMPLE, WEIGHT PER CENT			
2 to 1½ in....	25	...	...	...
1½ to 1 in....	25	50	...	...
1 to ¾ in.....	25	25	50	...
¾ to ½ in....	25	25	50	50
½ in. to No. 4.	...	...	...	50
	WEAR, PER CENT			
	13.0	11.0	9.3	12.0

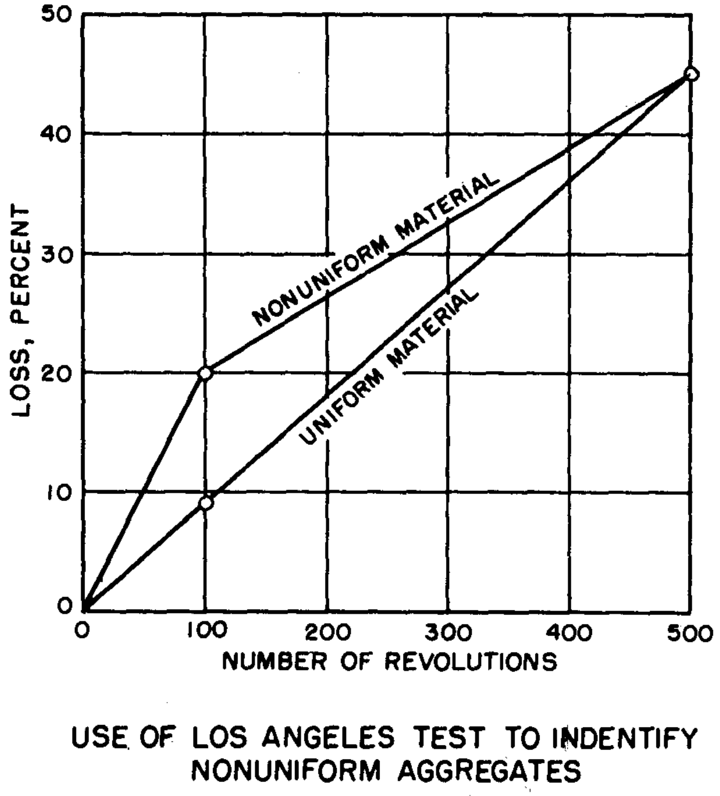

FIG. 1—Use of Los Angeles test to identify nonuniform aggregates.

five different gradings of the test sample, and only in exceptional cases are the losses for these different gradings approximately the same. Usually the losses vary with the different gradings, as is shown in Table 2 for a particular sample of gravel, with the differences between the losses frequently being much more marked. Unless the specifications for the material state the grading of the test sample to be used, the acceptance or rejection of the material may be based on the chance grading selected for the test.

Although the Deval test is still used by a number of State highway departments, it is time-consuming, requires meticulous preparation of test samples for rock, and gives test results that do not reflect significant differences in the materials tested. The application of the test for graded material is defective in that differences between the losses for the different gradings permitted are seldom acknowledged in specifications for aggregate.

Los Angeles Abrasion:

The Los Angeles abrasion test was

developed to overcome some of the defects found in the Deval test. The Los Angeles test is characterized by the quickness with which a sample of the aggregate may be tested, the high loss obtained, and the applicability of the method to all types of commonly used aggregates. With material of uniform quality, the loss varies directly with the number of revolutions of the machine. Consequently, a comparison of the losses after 100 and 500 revolutions should furnish some information of the presence of soft material in the aggregate. An example of the losses obtained for aggregates of uniform and nonuniform resistance to abrasion is given in Fig. 1.

In the construction of concrete pavements or bridge decks especially in locations where deicing salts will be used, it is believed most desirable to use coarse aggregates of uniform composition to prevent pitting or popouts of the concrete surface. In this respect, the ratio of the losses at 100 and 500 revolutions of the Los Angeles machine should be helpful in the selection of the coarse aggregates for use. Theoretically, the 100:500 ratio should be an excellent indication of the uniformity of the coarse aggregate tested. However, some research organizations state that they have been unable to find any association between this ratio and the performance of the aggregate in concrete [7]. As a number of state highway departments use and show this ratio in their specification requirements for coarse aggregates, it would appear that they have found the determination to be of value.

The Los Angeles test is described in ASTM Test for Resistance to Abrasion of Small Size Coarse Aggregate by the Use of the Los Angeles Machine (C 131 – 64 T) or Test for Resistance to Abrasion of Large Size Coarse Aggregate by Use of the Los Angeles Machine (C 535 – 64 T). It was adopted in 1939 and at that time provided for testing aggregate with either a 1½ or ¾ in. max size. Later two other sizes were added to permit the testing of finer sizes of aggregate, such as those used for sealing or covering bituminous pavements. In 1951 three more gradings were adopted to permit the testing of aggregates of large size prepared for use in trickling filters or base courses for pavements. By changes in the abrasive charge, the weight of the sample, or the length of the test, an attempt was made to obtain the same percentage of wear with each grading when a material of uniform quality was tested. With a reasonable margin for error, such uniformity was obtained for Gradings A, B, and C involving samples ranging in size from 1½ in. to the No. 4 sieve. No. 4 to No. 8 aggregate (Grading D) usually gave a loss varying only slightly from the three gradings mentioned above.

A reasonable number of tests by four laboratories have been made to determine whether Gradings E, F, and G for large pieces of aggregates would have about the same loss as Gradings A or B. A fair agreement was found in some instances but contrary data have also been obtained. On the basis of this lack of agreement and at the request by some organizations concerned principally with tests on large sizes of aggregates, Committees C-9 and D-4 have recommended that two methods be established for the Los Angeles abrasion test. Method C 131 – 64 T is to cover tests on aggregates passing the 1½-in. sieve; Method C 535 – 64 T is to cover tests on aggregates above the ¾-in. sieve. With these two methods for materials of distinctly different size, it should be less possible for absurd requirements for loss in the Los Angeles abrasion test to be made by persons unfamiliar with the methods.

The Los Angeles abrasion test is

considered by most material engineers to be superior to the Deval abrasion test for the determination of the quality of coarse aggregate. Wear or loss in the Los Angeles test appears to result from both impact and surface abrasion, with the first possibly causing more loss. Although impact does not appear to be a destructive influence of aggregates in concrete, the loss in the Los Angeles abrasion test has been correlated with the strength of concrete prepared with a wide variety of aggregates [*8*]. However, laboratory studies reported by Bloem and Gaynor failed to show a significant relation between the result of the Los Angeles abrasion test and the strength of concrete prepared with the aggregates [*7*].

When the Los Angeles test was considered for adoption as a standard, criticism was made of the use of both rounded and angular aggregates in the test. Doubt was expressed that with aggregates of the same quality the same percentages of wear would be obtained for materials composed of rounded or angular pieces. A number of different series of tests were made in the laboratory of the Bureau of Public Roads to investigate this. Tests were made on both plant-crushed stone and stone that had been broken into very nearly cubical shape. In the case of each material, the entire amount was mixed thoroughly and then divided into two equal portions. One portion of each stone was rounded in the Deval machine to simulate gravel. Three to six test specimens were prepared with each of the rounded materials as required for Grading A ($1\frac{1}{2}$ to $\frac{3}{8}$ in.) and an equal number with the unrounded stone. The results given in Table 3 were obtained. Although the values for the rounded and angular portions of each material are not the same, they agree sufficiently well to warrant testing crushed or uncrushed aggregate in the Los Angeles machine without making allowances for the shape of particle.

TABLE 3—EFFECT OF ROUNDED OR ANGULAR PIECES ON LOS ANGELES ABRASION TEST RESULTS METHOD C 131.

Material	Shape of Piece	Condition	Wear, per cent		
			Max	Min	Avg
Limestone No. 1	crushed	as received	33.8	33.2	33.4
		rounded	32.2	30.9	31.4
Limestone No. 2	cubical	angular	24.2	23.5	23.9
		rounded	22.7	21.9	22.4
Limestone No. 3	crushed	as received	27.6	26.8	27.1
		rounded	27.0	24.9	25.8
Sandstone No. 1	cubical	angular	63.7	61.8	62.8
		rounded	66.1	62.1	64.4

More variation in the Los Angeles per cent of wear is caused by the shape of the pieces tested, that is, whether the pieces are flat and elongated or cubical and spherical. As might be expected, flat and elongated pieces cause an increase in the loss. The ratio between the losses at 100 and 500 revolutions will be high if the aggregate contains undesirably thin or elongated pieces.

Questions of the relation between the losses for the Deval and Los Angeles tests have risen frequently and more may develop in the future. In the preparation of a summary of tests of coarse aggregates made in the laboratory of the Bureau of Public Roads [*9*], opportunity was afforded to collect data for samples tested by both methods. The results of abrasion tests for approximately 100 samples of gravel and over 700 samples of stone were obtained. The values for the Los Angeles test were restricted to samples tested with Gradings A or B. The results for the Deval test were limited to samples of rock tested in accordance with ASTM Method D 2 and to samples of gravel prepared and tested as required by Grading A of ASTM Method D 289.

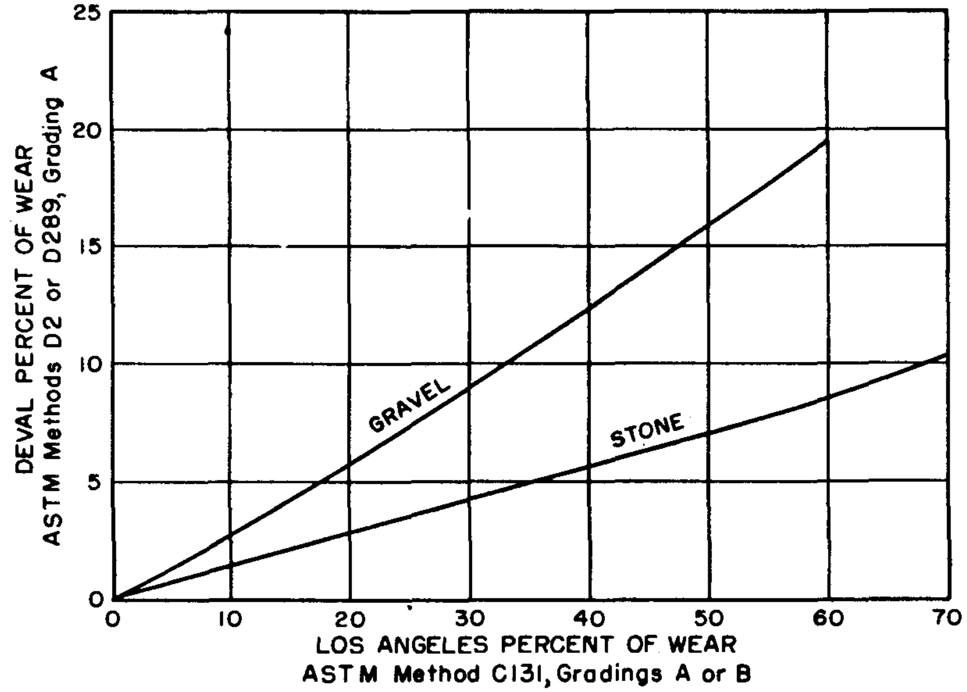

Fig. 2—Relation between results of abrasion tests of aggregates.

Curves showing these relations are plotted in Fig. 2. These curves differ somewhat from similar ones presented in an earlier paper [*10*], but many more samples are represented by the data given here.

Strength

Most specifications for aggregates require the material to be "strong." This does not necessarily imply the use of a test for compressive strength, but such a test is the one principally employed on natural materials. Because the test requires the use of a specimen of constant cross section, it is seldom applied to material other than ledge rock or large pieces of discrete aggregate from which specimens of regular shape may be prepared.

It is probable that specification writers have no firm opinions regarding the precise application of the word "strong." They desire that the pieces comprising the aggregate shall not be so weak or friable as to reduce the strength of the concrete. Concrete with a compressive strength of 6000 psi is quite common, and it might be assumed that aggregate should have at least this strength. The compression test is, however, inconvenient to make on aggregates composed of heterogeneous materials, and a test that can be made on the bulk aggregate should find favor. Although no such test is now in general use in this country, a test of this type is described in British

Standard 812:1951 [*11*]. In this test, $\frac{1}{2}$ to $\frac{3}{8}$-in. aggregate is placed in a steel cylinder, a steel plunger placed on the aggregate, and a load of about 3170 psi applied to the plunger. The amount of material broken to pass a No. 7 British Standard sieve (nominal opening 2.411 mm) is determined. The opening in this sieve is slightly larger than that in a No. 8 sieve defined in ASTM Specifications for Sieves for Testing Purposes (Wire Cloth Sieves, Round-Hole and Square-Hole Screens or Sieves, (E 11). The use of this British method for the determination of the quality of coarse aggregates for concrete may furnish valuable information.

TABLE 4—COMPRESSIVE STRENGTH OF TYPES OF ROCK COMMONLY USED AS CONCRETE AGGREGATES.

Type of Rock	Number of Samples[a]	Compressive Strength, psi		
		Avg[b]	After Deletion of Extremes[c]	
			Max	Min
Granite	278	26 200	37 300	16 600
Felsite	12	47 000	76 300	17 400
Trap	59	41 100	54 700	29 200
Limestone	241	23 000	34 900	13 500
Sandstone	79	19 000	34 800	6 400
Marble	34	16 900	35 400	7 400
Quartzite	26	36 500	61 300	18 000
Gneiss	36	21 300	34 100	13 600
Schist	31	24 600	43 100	13 200

[a] For most samples, the compressive strength is an average of 3 to 15 specimens.

[b] Average of all samples.

[c] Ten per cent of all samples tested, with highest or lowest values, have been deleted as not typical of the material.

Some limited aggregate crushing tests on railway ballast were made by the National Sand and Gravel Assn. and the National Slag Assn. [*12*]. In one case, 3000-g samples of ballast, graded uniformly from 1 in. to No. 4 sieve, were placed in a 6-in.-diameter steel cylinder and loaded to 3000 psi through a steel piston. The reduction in fineness modulus of each sample was determined. Crushing tests were made on $\frac{3}{4}$ to $\frac{1}{2}$-in. and $\frac{1}{2}$ to $\frac{3}{8}$-in. size samples of slag, each tested separately. A sample of 98 in.^{3} of compacted aggregate was weighed and divided into three equal parts for three tests on each size. The test sample was placed in a steel cylinder with a diameter of $5\frac{1}{2}$ in. and loaded through a steel piston to a total load of 11,000 lb. The sample was then sieved, and a crushing factor computed. This factor was the difference between the sum of the total percentage of material retained on the $\frac{1}{2}$-in., $\frac{3}{8}$-in., and No. 4 sieves before and after testing.

Information regarding the compressive strength of different types of rock will be found in many reference sources. The data presented seldom show the number of samples represented by the average value given, nor is the range of the compressive strength for normal samples shown. Although the information presented here can not be considered to represent an exhaustive compilation of test results, it is believed that the values given may be found of value in consideration of the quality of aggregate for concrete. These data were compiled from the results of routine tests made in the laboratory of the Bureau of Public Roads [*9*]. As shown in Table 4, an average value for all samples tested is included, in addition to a value for a possible maximum or minimum strength of rock of normal quality. These maximum and minimum values were obtained by considering that 10 per cent of all samples tested were too weathered for use in concrete and that a like number had abnormally high test strengths. For example, in the case of granite, the 28 samples with the lowest strengths and the 28 samples with the highest strengths were deleted. The maximum and minimum values then represent approximately 80 per cent of all samples of granite tested and are believed to show the range in strength for rock of normal quality.

CALIFORNIA TEST FOR QUALITY OF AGGREGATE

The heading given above is not entirely correct, but it furnishes an idea of the method of test to be described.

Virginia, as well as many of the Pacific Coast States, has found some rocks used in highway construction to degrade into plastic fines when under traffic and in the presence of water. Studies of similar aggregates and of methods for identifying them have been conducted by a number of the western states. Of the various methods developed, that devised by California appears to be the most successful. In this method, a sample of given size and weight of the aggregate under test is placed in a container with water and a small amount of calcium chloride solution, and agitated in a prescribed manner for 10 min. The solution with the abraded rock is then poured through a No. 200 sieve into a cylindrical container, agitated again, and the cylinder placed on a level surface to permit the nonflocculant fines to drop out of suspension. The height of this column of fines is then determined, and the durability factor for the aggregate computed by use of an empirical formula.

This method, as indicated above, was developed to determine aggregates which would break down into plastic fines, and such materials are foreign to the subject matter of this paper. However, Beaton [*13*] states that tests conducted by California have shown a surprising agreement between the results of this test and those of the accelerated (sulfate) soundness test. He states that aggregates with a high durability factor always have a low loss in the soundness test, and that aggregates with a low durability factor invariably have a high loss in the soundness test. He further states that tests for soundness may be required only when the durability factor has a median value, neither high nor low.

Any method of test which can furnish an evaluation of the resistance of aggregate to frost action quickly and more definitely than the sulfate soundness test should be welcomed by the materials engineer. The method developed by California appears to be of this type. Although that state has not made any suggestions of this nature, the use of this method to determine the over-all suitability of aggregates is proposed.

ELASTICITY

Nadai [*14*] states that the properties of natural rocks are to some degree comparable with those of artificial materials (metals). The granites, basalts, or other igneous rocks may be compared in their properties with cast metals. Such rocks have a high compressive strength. The same, but to a lesser degree, is true of compact sedimentary rocks such as sandstone or limestone.

Igneous and sedimentary rocks under ordinary compressive loads deform very little before they break. But with very long times, small differences between vertical and horizontal pressures in buried rocks may be sufficient to produce the observed folds in rock strata.

A universal property of crystalline and amorphous solids is their elasticity. A small but measurable portion of the total distortion of solid bodies under load is said to be of an elastic nature. On the release of force, these small deformations may disappear. The changes are proportional to the applied forces. Random orientation of crystalline components cancels out directional effects.

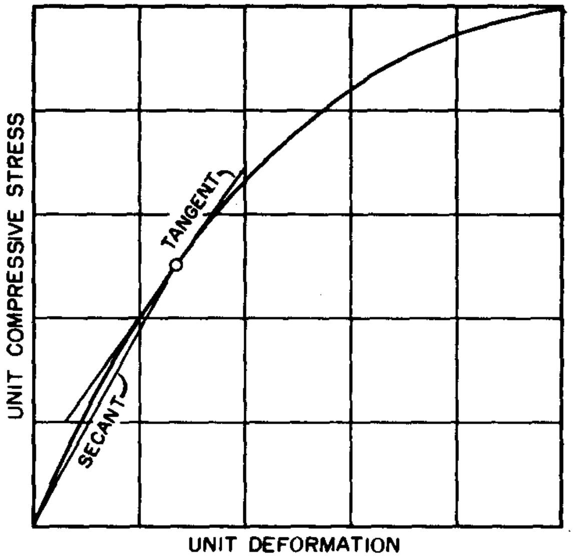

FIG. 3—Determination of modulus E (after Walker).

If a specimen of rock is subjected to an axial load, the height of the specimen will decrease by a certain amount, and the width of the specimen will increase. When the load, if not too great, is removed, the specimen will tend to return to its original dimensions. The specimen is then said to obey Hooke's law of proportionality of stress to strain. This may be, and usually is, expressed in different terms as the modulus of elasticity. If the load applied is within certain limits, Hooke's law may be stated as:

$$\frac{\text{Stress}}{\text{Strain}} = \text{a constant equal to modulus of elasticity}$$

Walker [*15*] has shown four methods for the determination of the modulus under static loading. These include the initial tangent modulus determined from a tangent to the stress-deformation curve near its beginning; the secant modulus determined from a line drawn from the origin of the curve to a point on the curve usually at one half of the total strength; the tangent modulus as determined from a line tangent to the curve usually at the same point as above; and a chord modulus computed from the slope of a line drawn between two points on the curve, possibly the limits for specified loadings. In Fig. 3, graphic conceptions of the secant and the tangent moduli are given.

In addition to direct axial loading of a specimen of regular form, the modulus of elasticity can be determined by the resonant frequency of vibration of specimens of regular shape (ASTM Test for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens (C 215)), or by the so-called supersonic method [*16*]. Here short-period pulses of high-velocity vibrations are sent into the material, and the time required for a pulse to traverse a known length is measured. In this method, the velocity of the ultrasonic wave, the density, and Poisson's ratio of the material must be known to determine the modulus of elasticity of the material tested.

The modulus of elasticity is used for a number of purposes, but in concrete engineering it is used mainly in the design of live-load bearing members. As the creep of the concrete in such members will affect their performance, an allowance for this is made usually by reducing the design modulus of elasticity [*17*]. Determination of the dynamic modulus of elasticity, measured from the resonant vibration frequency, is the most usual method for studies of the progress of disintegration of concrete subjected to freezing and thawing tests. The reduction of *E* (the modulus of elasticity) is related fairly well to the decrease in the transverse strength of the concrete specimen under test.

This transverse strength is much more sensitive to the deterioration of the concrete from frost action than is the compressive strength [*17*].

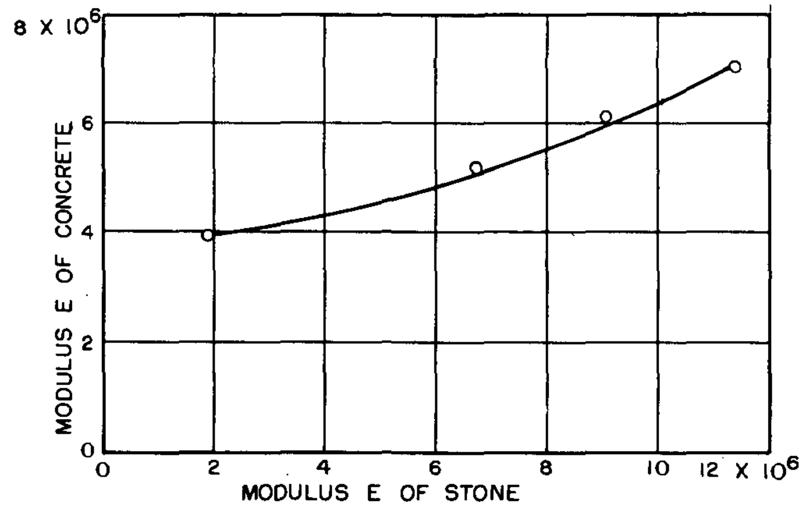

FIG. 4—Relation between modulus *E* of stone and concrete (LaRue's tests).

It has been found frequently enough to be accepted, that the modulus of elasticity of concrete has a fair relation with that of the coarse aggregate. The tempered term "fair relation" is used to indicate that some difficulties exist in the determinations of the two moduli. The nonuniformity of the rock, as mentioned above, difficulties in the available instrumentation for these determinations, and of more importance the lack of uniformity between the specimens of aggregate and the specimens of concrete, prevent obtaining truly representative values for these moduli unless very many determinations are made or the materials are of unusual uniformity.

Most of the available data regarding the modulus of elasticity has been obtained through use of the compression test, and only this method will be considered here. In this method, a specimen of uniform cross section is subjected to an axial load considerably less than the total load it will withstand, and the decrease in the length of the specimen determined. As shown in the discussion of Fig. 3, several different methods may be used to determine the modulus of elasticity. This value may be based on the initial tangent of the load-deformation curve, on the tangent modulus at some specified load, or on the secant modulus at some specified load. Due to the diversity of the values obtained with these several methods, there is little wonder that various investigators using different methods fail to agree with the findings for similar materials as mathematical errors do creep in, in spite of normal vigilance.

Of all the various studies of the effect of the *E* of aggregates on the *E* of concrete, one of the most interesting is a report by LaRue [*18*]. The author reported the results for only four aggregates, all limestones, but the results, as shown in Fig. 4, indicate a definite relation between the *E* of the aggregate and that of concrete prepared with these materials. LaRue concludes that the aggregate has a decided effect on the elastic properties of concrete, and that the relation of *E* of the aggregate to that of the concrete is not a linear function but may be expressed as an equation of exponential type for which the maximum value for *E* of concrete is approximately 6×10^6. However, Koenitzer [*19*] conducted studies of the elastic properties of stone and concrete using stone of the same type but from five different locations. He concluded that the elastic properties of the stone are not indicative of the results obtained for concrete. LaRue also states that no correlation between the compressive strength and the modulus of elasticity of concrete is evident from a study of the data he presented. This does not agree with the studies of other investigators. But at the best, these other studies show only a rough approximation between the two values.

Another very interesting report on the effect of the modulus of elasticity of aggregate on that of concrete has been presented by Hirsch [*20*]. Although this researcher chose to use in his study a number of aggregate materials which would not be used in construction concrete, he has been able to extend the scope of his determinations far beyond that which would be obtained with natural aggregates. Although his determinations are modified by the *E* of the cement paste matrix used, this report merits serious consideration by other researchers in concrete. Attention should also be given to the three discussions of this paper, and to Mr. Hirsch's closure [*21*].

Waddell [*22*] summarizes the question of *E* versus compressive strength by noting that a high-strength concrete usually has a higher elastic modulus than a weak concrete. He states that present thinking considers the static modulus as a function of the square root of the compressive strength, and that it is affected by the density of the concrete. In support of this latter statement, he shows curves for the modulus of elasticity of concretes prepared with natural and lightweight aggregates. These curves do show that for these concretes of the same ultimate strength, the modulus of elasticity of the lightweight concrete is about one half that of the concrete of normal weight. This is foreign to the general scope of this report but is furnished as a matter of interest.

In summation, it can be said that the modulus of elasticity of rock may be found to have a reasonable relation with that of concrete prepared with the aggregate in relatively few instances. In the one instance where a good relation is reported, it should be noted that all aggregates were limestones. No statement of their uniformity of structure and mineral composition is found, but it is assumed that these rocks are more homogeneous than is usual.

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Concrete Aggregates

THERMAL PROPERTIES

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The thermal properties of concrete aggregates are of greater or lesser concern to the user, depending upon the nature of the concrete structure and the exposure to which it is subjected. Examples of where knowledge of thermal properties are of greater concern are in the design of massive structures, such as dams, where thermal and volume stability are important and in lightweight concrete structures where insulating value is a primary factor. Except for thermal coefficient of expansion, the thermal properties of the aggregates used in normal concrete structures, such as office buildings, warehouses, pavements, and the like, are seldom considered. An understanding of the effects of the thermal properties of aggregates on the properties of concrete can have an effect on improving concrete quality and in some instances may well be mandatory.

The attention that has been given to the thermal properties of concrete aggregates is not as great as the amount of research and the volume of testing performed in connection with other properties of aggregate. This may be because the significance of thermal properties is not as apparent, and in many instances the effects do not appear to be as important as the effects of other properties of aggregates.

A discussion of the significance of the thermal characteristics of concrete aggregates is further complicated by the effect of the relationship between the thermal properties of the concrete as a whole and the thermal properties of the component materials. Insofar as possible the discussion in this paper is confined to the significance of tests of thermal properties of the aggregates; however, in some instances it has been necessary to discuss briefly the thermal properties of the concrete as affected by the aggregates. The significance of tests of thermal properties of concrete is discussed in another paper in this publication.[2]

The need for a better understanding of the effects of the thermal characteristics of aggregates, particularly the thermal coefficient of expansion on the durability of concrete, has been expressed by Allen [*1*],[3] Woolf [*2*], Scholer [*3*], and others.

The significance of other thermal properties has been given much less attention than has thermal expansion, as evidenced by the fact that the great majority of available references deal with thermal expansion. Undoubtedly the reason for this is that the thermal expansion of aggregate and hence of concrete is important from the standpoint of structural design in all concrete structures, whereas other thermal prop-

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[2] See paper by Mitchell, p. 202.

[3] The italic numbers in brackets refer to the list of references appended to this paper.

erties are usually of concern only in special instances, particularly in massive structures.

The thermal properties of aggregate referenced in the literature are thermal coefficient of expansion, specific heat, thermal conductivity, and thermal diffusivity, and they are discussed here in that order.

THERMAL COEFFICIENT OF EXPANSION

The earliest reference to work on this subject, included in this paper, is by Hallock [*4*] and is entitled, appropriately enough, "Preliminary Notes on the Coefficients of Thermal Expansion of Certain Rocks." Some aspects of this property, particularly with respect to the volume change of concrete as affected by the aggregates, are discussed in a paper on volume change in this publication.[4]

Numerical Values for Thermal Coefficients of Expansion:

Although values for the thermal coefficients of expansion of aggregates from specific locations are contained in nearly all of the references listed and are available from many other sources, Refs. [*5–15*] list more values than most of the others. It should be noted that Ref. [*15*] is concerned completely with the thermal coefficient of expansion of portland cement rather than aggregates. It is included because a discussion of the thermal properties of concrete aggregates would not be complete without some information on the thermal properties of the cements with which they are used.

An average value for the linear thermal coefficient of expansion of concrete may be taken as 5.5×10^{-6}/F (9.9×10^{-6}/C), but the range may be from about 3.2×10^{-6}/F (5.8×10^{-6}/C) to 7.8×10^{-6}/F (14.0×10^{-6}/C), depending upon the type and quantities of the aggregates, the mixture proportions, and other factors. Hydrated cement pastes may range from 6×10^{-6}/F (10.8×10^{-6}/C) to 9×10^{-6}/F (16.2×10^{-6}/C), and mortars from about 4.4×10^{-6}/F (7.9×10^{-6}/C) to 7.0×10^{-6}/F (12.6×10^{-6}/C). Mitchell [*5*] has found that at early ages or at certain critical saturations the linear thermal coefficient of expansion of cement pastes may be somewhat higher than those reported above. He reports values as high as 12.4×10^{-6}/F (22.3×10^{-6}/C) for some samples of neat cement specimens. He indicates that variation of thermal expansion of neat cement is related to the amount and characteristics of the gel and to an optimum (or pessimum) moisture condition producing maximum thermal expansion.

The linear thermal coefficient of expansion of common rocks ranges from about 0.5×10^{-6}/F (0.9×10^{-6}/C) to about 8.9×10^{-6}/F (16.0×10^{-6}/C) [*13*]. It should be kept in mind that some minerals exhibit anisotropic characteristics or the property of expanding more in one direction or parallel to one crystallographic axis than another. The most notable example is calcite, which has a linear thermal coefficient of expansion of 14.3×10^{-6}/F (25.8×10^{-6}/C) parallel to its *C* axis and -2.6×10^{-6}/F (-4.7×10^{-6}/C) perpendicular to this direction. Potash feldspars are another group of minerals exhibiting anisotropy. It is for this reason that the cubical expansion of rocks and minerals is not always directly related to the linear expansion, and this possibility should be kept in mind when investigating the thermal properties of aggregates.

Effect of Thermal Expansion:

There seems to be fairly general agreement that the thermal expansion of the aggregate has an effect on the durability

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[4] See paper by Washa, p. 189.

of concrete, particularly under severe exposure conditions or under rapid temperature changes. The agreement as to whether a high or a low coefficient of expansion is most desirable is not so general. In one of the earliest papers on the subject, Pearson [*16*], attributes a concrete failure to the use of an aggregate of low-thermal coefficient subject to severe frost action. Subsequent laboratory investigations described in the paper and in a subsequent paper [*17*] included freezing-and-thawing tests of concrete containing aggregates of both low and high thermal coefficients. The indications were that the concretes containing the aggregates of low thermal coefficient failed much more rapidly under the freezing cycle employed. Walker, Bloem, and Mullen [*18*], on the other hand, report the results of heating and cooling, concrete specimens over the temperature range of 40 to 140 F (4.4 to 60 C) at various rates. They found that "changes in temperature were destructive to the concrete with sudden changes in temperature being much more severe than slower ones; and concretes having higher coefficients of expansion were less resistant to temperature changes than concretes with lower coefficients." It was also determined that the thermal coefficients of expansion of concrete and mortar containing different aggregates varied approximately in proportion to the thermal coefficient and quantity of aggregate in the mixture. The anomalous results obtained by Pearson and by Walker, Bloem, and Mullen perhaps may be explained by the fact that the concrete in Pearson's studies was subjected to temperatures below the freezing point, whereas that used by the other workers was not. The above is conjecture but is somewhat supported by Koenitzer [*19*], who obtained coefficients of linear expansion of several concretes and the aggregates used in them over temperature ranges of 9 to 80 F (−12.8 to 26.7 C), 85 to 190 F (29.4 to 88 C), and 9 to 190 F (−12.8 to 88 C), in both moist and dry conditions. One of Koenitzer's conclusions was that the elastic and thermal expansion properties for any one material vary with the conditions of test, the greatest variation being caused by freezing.

The U.S. Bureau of Reclamation's experience with aggregate at Grand Coulee Dam showed an extremely high resistance to freezing and thawing. This aggregate was predominately basalt with a thermal expansion of about 4×10^{-6}/F (7.2×10^{-6}/C).[5] The U.S. Bureau of Reclamation also found [*6*] in the Kansas-Nebraska area that replacement of part of the aggregate with limestone, which had a thermal expansion averaging 2.5×10^{-6}/F (4.5×10^{-6}/C), greatly increased the durability of the concrete in which it was used. It should be noted that the addition of the limestone was for the purpose of inhibiting cement-aggregate reaction, and the improvement in durability probably was primarily because of the inhibition of this reaction, but the low thermal expansion apparently introduced no adverse effects.

Callan [*20,21*] has statistically analyzed 78 combinations of aggregate in concrete with respect to durability in freezing and thawing and differences in thermal expansion between the coarse aggregate and the mortar. He concludes that, where the difference between coefficients of expansion of coarse aggregate and mortar is large, the durability of the concrete may be considerably lower than would be predicted from the results of the usual acceptance tests. Where the differences between these coefficients exceeds 3.0×10^{-6}/F (5.4×10^{-6}/C), caution should be used in the selection of the aggregate combination

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[5] L. J. Mitchell, private communication.

for highly durable concrete. Walker, Bloem, and Mullen [*18*] found no relationship between resistance of concrete to temperature changes and differences between thermal coefficients of aggregates and mortar. Again Callan's work is based on studies over the freezing-and-thawing range, whereas the results of the other workers were based on experiments above the freezing temperature. Swenson and Chaly [*22*] include some discussion of the effect of thermal coefficient of expansion of the aggregates on concrete durability and caution against the possible deleterious effects if large differences are observed. Smith [*23*] presents calculations to indicate the potential magnitude of physical incompatibility of the matrix and aggregate in concrete based on differences in thermal expansion. Kennedy and Mather [*24*], in attempting to correlate laboratory-accelerated freezing and thawing with natural weathering at Treat Island, Me., state among other conclusions that, while there appears to be a correlation between the resistance of concrete to freezing and thawing and differences in thermal expansion between the coarse aggregate and the mortar, the correlation is probably usually of lesser importance than other characteristics of the concrete.

The above discussion of potential incompatibility between the matrix and the aggregate of concrete has serious implications, although, as yet, the proven correlation in actual field experience is not strong. It would seem that one should be alert to the possibility of difficulty under unusual situations but that sound engineering judgment would not dictate unusual precautions as being necessary with normal concrete and normal aggregate.

All of the above discussion has dealt with the effects of thermal expansion of aggregates over temperature ranges that can occur under natural exposure conditions. Much less is known about these effects under more extreme ranges as, for example, under the conditions that may exist in burning buildings. The U.S. Bureau of Reclamation reports [*14*] that at a temperature of 1063 F (572.7 C) quartz changes state and suddenly expands 0.85 per cent, usually producing a disruptive effect at the surface of concrete in which it is used. Endell [*25*] has reported the results of experiments to determine the structural and expansion changes of concrete aggregates with temperatures up to 2192 F (1200 C). These are considered to be highly specialized conditions and are not discussed further here, but are discussed further in another paper in this publication.[6]

Methods of Determining Thermal Expansion of Aggregates:

Several ingenious methods have been developed for determining the thermal coefficient of expansion of coarse aggregate. The majority of the test methods are based on the measurement of linear expansion over a temperature range. This range is usually 100 F (55 C) or more because the change in unit length per degree is extremely small, and the multiplication of the change over a substantial temperature range greatly increases the facility and precision of the measurements. Likewise, the longer the specimen the greater is the accuracy and precision of the determination. However, the size of a representative specimen that can be obtained from a coarse aggregate rapidly approaches a practical maximum. Except in the case of a crushed aggregate of sufficient uniformity to permit obtaining a larger specimen that will be representative of the sand sizes, this means of multiplying the change in unit length is not available for determining the linear expansion of

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[6] See paper by Petersen, p. 290.

fine aggregate. Another device that has been used to obtain additional multiplication of the length change is the optical lever.

The method described by Willis and DeReus [*26*] is an example of measurements made over a considerable temperature range with the use of an optical lever. The specimens were 1-in. (2.54-cm) diameter cores, 2 in. (5 cm) long, drilled from the aggregate specimens to be tested and placed in a controlled-temperature oil bath with a range of 37 ± 3 F (2.78 ± 1.7 C) to 140 ± 5 F (60 ± 2.8 C). The vertical movement of the specimen as the temperature was varied was measured by reading the image reflected by the mirror of the optical lever, having a 1-in. (2.54-cm) lever arm, on a vertical scale placed 20 ft (6.1 m) from the mirror, by means of a precise level. It is reported that consideration of the possible errors involved in the measurements indicates that the calculated coefficients are probably accurate to $\pm 2.0 \times 10^{-7}$/F (3.6×10^{-7}/C).

Another method is the interferometer method described by Merritt [*27*] and modified by Saunders [*28*]. Detailed descriptions of the apparatus, the preparation of specimens, and the test procedure are also given by Johnson and Parsons [*8*]. A third method for determining the thermal coefficient of expansion of coarse aggregate is that developed by the Corps of Engineers [*21,29*] in which an SR-4 strain gage is bonded to a prepared piece of aggregate and readings taken over a temperature range of 35 to 135 F (1.7 to 57.2 C). This method requires that the piece of aggregate be sliced in three mutually perpendicular directions, two of these directions to lie in the major structural plane of the rock, if such plane can be located. The strain gages are then mounted so as to measure strain in each of the three directions. The purpose of this requirement is to determine if anisotropy or preferred crystal orientation exist.

Also the current method used by the U.S. Bureau of Reclamation and described by Mitchell [*5*] is available. This method employs specimens from 1 to 3 in. (2.54 to 7.62 cm) in size coated with wax and held in fulcrum-type extensometer frames. Measurements are made with electromagnetic strain gages with electronic indicators while the specimen is immersed in a circulating ethylene glycol solution held at the desired temperature. The details of the method are well described in the reference.

Because of the size and usually heterogeneous nature of fine aggregate, none of the above methods are readily adaptable to the determination of the thermal coefficient of expansion of this type of material. The usual approach has been to determine the linear expansion of mortar bars containing the fine aggregate. The results obtained include, of course, the effect of the length change contributed by the cement. Verbeck and Hass [*30*] have developed a dilatometer method for determining the thermal coefficient of expansion which is particularly adaptable for use with fine aggregate. The method determines the cubical thermal coefficient of expansion from which the linear expansion may be calculated. The apparatus consists of a 1-liter dilatometer flask to which is attached a capillary-bulb arrangement containing electrical contacts spaced over a calibrated volume. The flask is filled with aggregate and water and the apparatus allowed to come to equilibrium at one of the controlling electrical contacts. The equilibrium temperature is noted and the procedure repeated at the other electrical contact. After proper calibration, the only measurements required are the weight of the water placed in the flask and the temperature needed to produce an expansion equivalent to the volume between the electrical contacts. This method offers a tool for determining directly the average thermal expansion properties of the smaller aggregate sizes that has not been available heretofore.

THERMAL CONDUCTIVITY, THERMAL DIFFUSIVITY, AND SPECIFIC HEAT

Thermal conductivity, thermal diffusivity, and specific heat are largely interrelated, and all three are normally determined only for concrete as used in massive structures. Reference [*31*] indicates the application of these data in connection with computing concrete placement temperatures and designing cooling systems, and in other thermal calculations aimed at reducing thermal volume change and thus cracking in large dams. The same type of measurements and calculations would equally apply to other massive structures. Thermal conductivity is also of importance in lightweight concrete for insulation purposes. It has been indicated by some [*32–34*] that thermal diffusivity may have an important effect on concrete durability.

Thermal conductivity, measured as the rate of heat flow through a body of unit thickness and unit area with a unit temperature difference between two surfaces, is normally expressed for calculations for concrete in the English system as Btu/ft-hr-deg F or in the metric system as cal gm/cm-hr-deg C.[7]

Thermal diffusivity is defined as the thermal conductivity divided by the specific heat and density and is a physical property of the material which determines the time rate of change of temperature of any point within a body. Its English units are square feet per hour and metric units are square centimeters per hour.[7]

Specific heat is the amount of heat required to raise the temperature of a unit mass of material one unit of temperature. Its English units are Btu per lb per deg F, and metric units are cal, gm per gram per deg C.[7]

Thermal Conductivity of Lightweight Aggregates:

One of the more useful properties of lightweight concrete is its low thermal conductivity. Since thermal conductivity usually varies directly with density, aggregates of low density produce concrete of lower conductivity. For the same reason, as indicated by Tyner [*35*], moisture has a tremendous effect on thermal conductivity. He reports that in a 1:5 mix of Florida limerock concrete an increase of moisture from 0 to 5 per cent increases the thermal conductivity by 23 per cent and an increase from 0 to 10 per cent increases the conductivity by 46 per cent. Davis and Kelly [*36*] also state that "the presence of a small amount of moisture in the interior of a lightweight concrete greatly increases its thermal conductivity; hence, under conditions of continuous or intermittent exposure to moisture, if a high degree of insulation is desired, an aggregate (and concrete) of relatively low absorption should be used." Kluge, Sparks, and Tuma [*37*] and Price and Cordon [*38*] also have found pronounced reductions in the thermal conductivity of concrete containing lightweight aggregate, but they indicate that the reduction seems to be influenced more by the reduction in the density of the concrete than by the characteristics of the aggregate.

While the relationship of reduced

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[7] The metric units shown should be considered as examples only. Users of the metric system should be aware that there are several metric units which may be used alternatively, for example, there are the cal gm, the cal kg, the cal gm (IT), and others. In the interpretation or comparison of thermal values the user should be sure of the particular units used in the calculations.

thermal conductivity with reduced density holds generally for normal and lightweight aggregate, it may not hold for heavyweight aggregates. Investigations of barite for use in concrete for radiation shielding by Witte and Backstrom [*39*] indicate its thermal conducductivity to be somewhat lower than normal concrete aggregate.

Thermal Diffusivity:

Relatively recent investigations have indicated that the thermal diffusivity of the aggregate may have an influence on the durability of the concrete in which it is used. Thomson [*32*] states that for a given body with specified boundary conditions the thermal stresses depend on certain physical properties of the materials. In a homogeneous body such physical properties as thermal conductivity, specific heat, and the density of the material, influence the temperature distribution and the thermal stresses during the transient period only in a certain combination known as the thermal diffusivity. If in a mixture such as concrete the thermal diffusivities and conductivities are the same for each material, the body can be thought of as being thermally homogeneous. Since a difference in diffusivities would result in different rates of diffusion of heat through the aggregate and cement, it is believed that such a combination would result in higher thermal stresses than those existing in homogeneous bodies. Nothstine [*33*] and Weiner [*34*] have reported the results of their approach to the problem. Weiner's work was instigated by the failure of a gravel concrete, exposed to natural freezing and thawing accompanied by thermal shock and characterized by bond failure and internal expansion. He attributes the failure to the relatively high thermal coefficient of expansion of the concrete, which is responsible for surface stress, and to the diffusivity of the gravel which, being higher than the mortar, responds more quickly to temperature changes, resulting in differential volume change. Fox and Dolch [*4*] in an investigation of four limestones found a large change in the thermal diffusivity with a relatively small degree of saturation. The increases in diffusivity ranged from 20 to 59 per cent for saturations of less than 5 per cent. The authors of the references cited essentially agreed that the thermal diffusivity of the aggregates apparently has an effect on the durability of concrete but that further work is needed to determine the significance of the effect and to find a practical means for applying this knowledge to the improvement of concrete durability.

Specific Heat of Aggregates:

Specific heat is of considerable importance in connection with the calculations involved in the control of placement temperatures and the limiting of thermal volume change of mass concrete. The specific heat of the aggregate contributes materially to the specific heat of the concrete [*31*].

Methods of Test for Conductivity, Diffusivity, and Specific Heat:

Methods are available for the direct determination of thermal conductivity, thermal diffusivity, and specific heat. However, as a matter of practical convenience, it is customary to determine diffusivity and specific heat and calculate conductivity or to determine conductivity and specific heat and calculate diffusivity. This is possible because the formula includes all three values, and knowing the values for any two, and the density, permits solving the equation for the unknown property. The formula is:

$$k = h\,c\,p$$

where:
k = thermal conductivity in Btu/ft-hr-deg F,
h = thermal diffusivity in ft^2/hr,
c = specific heat in Btu/lb-deg F, and
p = density in lb/ft,^{3}
(or appropriate metric units—see footnote 7).

Whether conductivity is determined directly and diffusivity calculated, or vice versa, is largely a matter of the most convenient equipment setup available and the preference of the laboratory doing the work. Since the conductivity of a mixture is not an additive function of the constituents [*41*], the conductivity and diffusivity of aggregate cannot be obtained indirectly by determining these properties of the concrete and mortar. Some of the test methods included in the references to this paper are based on determinations made on concrete specimens, but in most cases they are equally applicable to use with aggregates if properly modified with respect to specimen size and shape.

The Corps of Engineers [*29*], Thomson [*32*], and Fox and Dolch [*40*] describe methods for the direct determination of diffusivity of stone and concrete. All of the methods depend basically on obtaining time-temperature differential curves between the temperatures at the center and the surface of a specimen by starting at essentially equilibrium temperature, then changing the surface temperature, and plotting the time-temperature curve until equilibrium conditions are obtained at the new surface temperature. Depending upon the degree of accuracy desired, refinements can be made by grinding the specimen to a sphere, for example, and by refining the instrumentation. The determination is by no means routine in nature and requires careful experimentation, precise equipment, and a capable operator.

Thermal conductivity when measured directly is determined by ASTM Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (C 177) or similar methods [*35,42*]. Since the equipment is designed for use with a flat specimen either square or round, it is not easily adapted to precise determinations of the conductivity of such materials as concrete aggregates. For this reason it is generally considered better to determine diffusivity and calculate conductivity for such materials. The Bureau of Reclamation [*43*] has developed a method for the determination of the thermal conductivity of concrete by the use of an 8 by 16-in. (20.3 by 40.6-cm) hollow cylindrical concrete specimen. This procedure could probably be used for coarse aggregate provided a specimen of the required size and shape could be fabricated from a large rock specimen.

The specific heat of aggregate is usually determined by a procedure known as the method of mixtures [*29*]. It is a calorimetric procedure wherein the net heat required to raise the temperature of a specimen of known weight a given amount is measured.

References [*44–52*] provide additional background information on the theory and mathematics of thermal tests. Other references may be found appended to many of the references cited here.

CONCLUSIONS

Test methods are available which when used with proper attention to procedure, specimen size and shape, instrumentation, and technique are entirely adequate for the determination of the thermal properties of aggregates.

There appears to be no doubt that the thermal properties of the aggregates, particularly thermal expansion, have an effect on the durability and other qualities of concrete. Investigations reported to date do not present a clear-cut picture of the effects that might be expected, and some aspects of the problem are controversial. The ultimate solution must be based on the performance of aggregates of known thermal properties in concrete, and, as is normal in this field of investigation, the major difficulty is to separate the effects of the thermal properties of the aggregates from the numerous other variables existing in the concrete. There is a real need for additional research work on the subject, both to resolve existing controversy and to improve concrete further as a construction material.

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Concrete Aggregates

CHEMICAL REACTIONS

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ASTM Tentative Definition of the Term Aggregate (C 58 – 28 T), defined aggregates as follows: "Aggregate—In the case of materials of construction, designated inert materials which when bound together into a conglomerated matrix forms concrete, mastic, mortar, plaster, etc."

It has long been recognized that aggregates might contain impurities that are not inert in portland cement concrete. For example, ASTM Standard Method of Test for Organic Impurities in Sands for Concrete (C 40), was published as a tentative method in 1921. Actually, an aggregate may be inert in one matrix, for example, plaster of Paris, and reactive in another, such as, portland cement paste. Over the years concrete technologists have found that it is unsafe to assume that crushed stone and natural aggregates, from sources without service records, will prove to be inert in portland cement concrete. Lerch [*1*][2] has reviewed the early efforts to develop test methods for the purpose of evaluating the potential reactivity of aggregates in portland cement concrete. These efforts resulted in the following methods.

1. C 227, Standard Method of Test for Potential Alkali Reactivity of Cement Aggregate Combinations (Mortar Bar Method).
2. C 289, Standard Method of Test for Potential Reactivity of Aggregates (Chemical Method).
3. C 295, Recommended Practice for Petrographic Examination of Aggregates for Concrete.
4. C 342, Standard Method of Test for Potential Volume Change of Cement-Aggregate Combinations.

Section 4(*c*) of ASTM Standard Specifications for Concrete Aggregates (C 33) states, "Fine aggregate for use in concrete that will be subject to wetting, extended exposure to humid atmosphere, or contact with moist ground shall not contain any materials that are deleteriously reactive with the alkalies in the cement in an amount sufficient to cause excessive expansion of mortar or concrete, except that if such materials are present in injurious amounts, the fine aggregate may be used with a cement containing less than 0.6 per cent alkalies calculated as sodium oxide or with the addition of a material that has been shown to prevent harmful expansions due to the alkali-aggregate reaction." Paragraph 8(*b*) contains the same statement applied to coarse aggregate. These statements are followed by a note that suggests that results of tests made in accordance with the four test methods listed above will assist in evaluating aggregates for potential reactivity in concrete. The literature pertaining to the reactions of aggregates in concrete is

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

extensive and has been reviewed rather thoroughly by several authors [*1–6*].

REACTIONS OF AGGREGATES IN CONCRETE

Normally, applied chemical research on a given problem consists of gathering data, formulating and evaluating hypotheses, rejecting unsound hypotheses, and finally reaching an understanding of the reactions involved with the result that competent technologists can predict results with reasonable accuracy and outline relatively simple tests to evaluate their predictions. The research with some types of reactive aggregates in portland cement concrete appears to have reached this final stage.

It seems safe to state that at atmospheric temperatures, which may range from well below the freezing point to somewhat below the boiling point of water, none of the solids found in portland cement concrete can react as solids with any other of the solids at rates sufficiently rapid to be of importance in the behavior of the concrete in service. It is important to note that the word "inert" in the above definition of aggregate must be interpreted as meaning that, if the aggregate regarded as inert does react with its environment, the rates of the reactions are so low as to render the reactions harmless in the performance of the concrete. Lerch [*1*] has pointed out that a limited chemical reaction between the aggregate and components of the paste is believed to be beneficial with respect to the bond between the aggregate and the paste and accordingly beneficial from the standpoint of strength. Mather and Mielenz [*7*] suggested that epitaxy may be responsible for improving the quality of concrete made with sand-gravel aggregates when limestone is used as a replacement for some of the sand-gravel aggregate. In this case, it is suggested that the calcite crystals of the limestone act as nuclei for the crystallization of some of the hydrated cement minerals and accordingly cause the formation of a bond between the aggregate and paste.

Since the solids cannot react with each other as solids, cement technologists are concerned only with reactions in which either an aqueous solution or water vapor is one of the reactants. In normal portland cement concrete, the liquid phase, for approximately the first 24 hr, is a solution consisting almost entirely of alkali and calcium sulfates and hydroxides. Chemically, this is an alkaline solution whose most reactive component is the hydroxyl ion. The hydroxyl ion concentration of this solution, except possibly in badly deteriorated concrete, can never be less than that of a saturated solution of calcium hydroxide. It can exceed this, and the amount by which it does exceed this concentration depends upon the alkali content of the cement and possibly, in some cases, on alkalies supplied to the solution by the aggregates or the ambient environment, that is, soil, water, and air.

In the development of ASTM Specification for Portland Cement (C 150) and ASTM Specification for Concrete Aggregates (C 33), it was recognized that the reactive component of the alkalies was the hydroxyl ion produced when the alkalies of the cement were released to the liquid phase of the cement paste and formed hydroxides. Because of this recognition and since one molecular weight of potassium oxide yields the same number of hydroxyl ions to the solution as does one molecular weight of sodium oxide, ASTM Specification C 150 in Note 3 expresses the total alkalies of the cement in terms of the equivalent amount of sodium oxide.

Some amount of air may be entrained by the cement paste or trapped in the pores of the aggregate during the mixing and placing operations. Also, when concrete goes through cycles of wetting and drying, the water taken up by the concrete almost certainly carries some air into the concrete. Any carbon dioxide in the air is precipitated quickly as calcium carbonate and is not available for reaction with the aggregates. However, the oxygen of the air is available for reaction with components of the aggregates and with steel reinforcing or other steel in the concrete. Hence, in addition to the ions and molecules listed above, molecular oxygen can also be a reactive component of the liquid phase of portland cement concrete.

It appears to be well established [*8*,*9*] that there are three types of chemical reactions that can cause expansions in portland cement concrete. These are as follows:

1. *Solid-water vapor reactions*—reactions in which a solid takes up water vapor to form a solid-reaction product. This type of reaction is represented later by Eq 1.
2. *Solid-liquid reactions*—reactions in which a solid reacts with ions or molecules of water or with ions or molecules dissolved in the aqueous solution or both to yield a solid reaction product. This type of reaction is represented later by Eq 2.
3. *Reactions which give rise to hydraulic pressure*—reactions in which a solid reacts with ions or molecules of water vapor or of the aqueous solution to yield a solid reaction product which when it dissolves yields molecules and possibly ions that are too large to migrate through the pores of the hardened cement paste and, accordingly, through osmosis create hydraulic pressure at the site of the reaction. There is a possibility that the solid reaction product reacts with calcium hydroxide from the liquid phase to form a product that acts as the semipermeable membrane instead of the hardened cement paste. This type of reaction is discussed later under Eq 2.

These appear to be the only types of reactions involving aggregates that can cause expansions in concrete. However, in the case of organic materials, such as coatings on sand grains, the alkaline solution may react with this coating to produce a reaction product that either interferes with the setting and hardening of the cement paste or entrains excessive amounts of air [*6*]. Likewise, it is possible for one or more of the reaction products of a reaction between an aggregate and the liquid phase of the concrete to migrate from the site of the reaction and react deleteriously with components of the cement paste. For example, a sulfide mineral in the aggregate may react with oxygen and calcium hydroxide to form calcium sulfate. The latter may then migrate into the cement paste and react with the aluminates to form calcium sulfoaluminates.

REACTIVE COMPONENTS OF AGGREGATES

Extensive searches have been made for materials in aggregates that will react with alkaline solutions similar in composition to the liquid phase of portland cement concrete [*4*,*5*]. Some of these materials may also react with pure water or with water vapor. It seems likely that the classes of compounds that might be found in aggregates and are capable of reacting in concrete have been identified and described. It should be possible, therefore, to describe all of the possible deleterious reactions between aggregates and the liquid phase of concrete that are likely to be encountered in service. This will be attempted in the following pages. This discussion will not be limited to natural aggregates but will include materials such as blast furnace slags and expanded clays and shales. It appears that the deleteriously reactive components of aggregates, other than organic impurities, are found in the following classes of materials, oxides, sulfides, glasses, calcium sulfate, zeolites, clays and certain dolomitic limestones. The reactions of each of these materials will be discussed.

Oxides:

The reactive oxides that may be found in aggregates probably are limited to those of calcium, magnesium, silicon, and iron: CaO, MgO, SiO_2, and FeO. The first two and possibly the last will react with water or water vapor to give hydrates as illustrated in Eq 1 for the reaction of CaO with water.

$$CaO + H_2O = Ca(OH)_2 \quad \ldots.(1)$$

CaO probably never occurs as a component of natural aggregates, except through contamination, but it has been observed in blast furnace slags and in expanded shale [*6*]. On the other hand, MgO as periclase [*6*] does occur in some metamorphic silica-poor dolomitic limestones and some dolomites where dolomite has been decomposed to calcite and periclase near igneous contacts. However, rocks containing periclase are relatively uncommon.

Since these oxides may be components of aggregates and since they can react with water without assistance from the alkalies from the cement, it seems that the statement quoted in the introduction from ASTM C 33 should be modified to read as follows: "Shall not contain any materials that are deleteriously reactive with the liquid phase of concrete." It has been shown that some aggregates and pozzolanic materials [*10*] can release alkalies to solutions of calcium hydroxide. The modified statement would recognize that water, by itself, might react with some components of aggregates. Also, it would recognize that deleterious alkalies might be derived from other components of the concrete than the cement. In this connection it should be mentioned that clays and zeolites can exchange alkali ions for calcium ions and, accordingly, if these materials are present in the aggregate, they can furnish alkalies to the liquid phase of the concrete.

Silica: SiO_2 exists in several crystalline modifications. The three of interest to the concrete technologist are quartz, cristobalite, and tridymite. It also exists in nature as a more or less amorphous partially hydrated material known as opal. Although cristobalite and tridymite have been considered as forms of SiO_2, and although they may be so classified for convenience, the natural materials and many of the synthetic examples show considerable variation in composition [*11–13*] supporting the conclusion that neither material can exist without the presence of foreign ions, usually including sodium, calcium, and aluminum. Many opals of inorganic origin consist of irregular alternations of cristobalite- and tridymite-layers interrupted by foreign ions. Some organic opals are entirely amorphous to X-rays.

Chalcedony is a microcrystalline to cryptocrystalline fibrous form of quartz. In some rocks quartz crystals are fractured intensely, strained or filled with inclusions or both. All these forms of silica, except well crystallized and relatively large crystals of quartz, are classed as potentially reactive in the liquid phase of portland cement concrete [*4*]. Silica forms a number of alkali silicates, some of which are rather highly hydrated. For example, Vail [*14*] reports a crystalline silicate of the composition $Na_2SiO_3 \cdot 9H_2O$. This formula could also be written as $Na_2H_2SiO_4 \cdot 8H_2O$. Using this silicate as an example, the reaction of silica with sodium hydroxide of the liquid phase of the cement paste may be illustrated by Eq 2:

$$SiO_2 + 2NaOH + \text{water} = Na_2H_2SiO_4 \cdot 8H_2O \quad (2)$$

The rate of this reaction will increase with the degree of instability of the SiO_2, increasing surface area of the SiO_2, increasing temperature, and increasing hydroxyl ion concentration of the ambient solution. Opal, which may be characterized as a solidified water-containing gel, probably is the least stable of the materials classified as silica, and it has been found to be very reactive in concrete. It occurs in basaltic lava and in shale. It also occurs as coatings on pebbles of sedimentary rocks, as the cementing medium in some sandstones and siltstones, and as interstial material in limestone formations, sometimes together with flint and chert.

Silica reacts with alkali hydroxides by a solid-liquid reaction [*15,16*] which enlarges the particle and causes it to exert pressure on the confining cement paste. Furthermore, the product formed by this reaction continues to take up water to form a gel-like product which tends to become more and more fluid. This take-up of water causes the fluid-like product to exert pressure on the confining cement paste by what might be classed as hydraulic pressure.

Ferrous oxide: FeO probably is rarely, if ever, found in natural aggregates. However, it might occur in slags, and it would be expected to react with water as illustrated in Eq 1 for CaO.

Sulfides:

Iron is found in many rocks in the form of ferrous sulfides such as marcasite (FeS_2), pyrite (FeS_2), and pyrrhotite ($Fe_{x-1}S_x$). These are ferrous sulfides in which the sulfur is stoichiometrically in excess of the iron. In the presence of oxygen and calcium hydroxide, FeS_2 could react as illustrated in Eq 3.

$$FeS_2 + O_2 + 2Ca(OH)_2 + \text{water} = Fe(OH)_3 + 2CaSO_4 \cdot 2H_2O \quad (3)$$

These reactions might occur as solid-liquid reactions and cause expansion of the concrete. The calcium sulfate might dissolve and migrate into the cement paste and react with calcium aluminate to produce calcium sulfoaluminate and cause expansion.

It appears that distress in concrete caused by ferrous sulfide-bearing aggregates has been limited to popouts where the offending aggregate was close to the surface of the concrete. This probably shows that the oxygen required by the reaction, which has to be obtained from the air, does not penetrate deeply into good concrete. This is another case in which the reactive component of the aggregate does not require assistance for its reaction in the concrete from alkali hydroxides released from the cement. The calcium hydroxide required for the reaction illustrated in Eq 3 is furnished by the reactions of the cement minerals with water.

Air-cooled blast-furnace slags usually contain significant amounts of calcium sulfide (CaS). This mineral could react in concrete as shown in Eq 4.

$$CaS + 2O_2 + \text{water} = 2CaSO_4 \cdot 2H_2O \quad (4)$$

It is believed that this could be a solid-liquid reaction and, accordingly, could be a source of expansion in concrete. Also the calcium sulfate formed in this reaction could migrate into the cement paste and react with the aluminates to produce calcium sulfoaluminates. CaS is somewhat soluble in solutions of alkaii hydroxides. Hence, it might dissolve as such and diffuse into the liquid phase of the cement paste and there react with oxygen to form sulfates which, in turn, would react with the aluminates. It appears [5] that no deterioration of concrete has been traced to any reaction of the CaS from blast furnace slag. This may be due to the limited availability of oxygen in good concrete.

Glasses:

Glasses, either natural or synthetic, are unstable systems and, in many instances, are attacked readily by alkaline solutions. The reaction, in this case, is probably very similiar to those described for silica. Since all of the natural glasses contain some alkalies, Na_2O and K_2O, the reaction with water or hydroxyl ion tends to form an alkali silicate, as illustrated in Eq 2 for SiO_2. The first water is taken up as a solid-liquid reaction which causes the glass particle to expand. The alkali silicate formed by this reaction continues to take up alkali ions and water to form a gel-like mass similiar to that formed when opal or other forms of reactive silica react with alkalies. Hence, the expansions produced by glasses are first produced by a solid-liquid reaction, and then this may be augmented by hydraulic pressure.

Calcium Sulfate:

Gypsum, $CaSO_4 \cdot 2H_2O$, if present in clays and shales used in the production of expanded clay and shale aggregates, is likely to be dehydrated to anhydrite, $CaSO_4$, during the expanding process. Anhydrite and gypsum may also be present in natural aggregates. If anhydrite is present in relatively large particles and relatively large amounts, it appears likely that it could cause expansion in concrete during rehydration to gypsum as illustrated in Eq 5

$$CaSO_4 + 2H_2O = CaSO_4 \cdot 2H_2O \ldots\ldots (5)$$

It is well known that gypsum in cements in excess of an optimum amount tends to cause excessive expansions in concrete by reaction with the calcium aluminates. Hence, if the aggregates contain more than relatively small amounts of either anhydrite or gypsum, this calcium sulfate coud react deleteriously with the cement paste.

Zeolites:

Zeolites are secondary minerals formed by the hydration of alkali and alkaline earth aluminum silicates. Their crystals consist of an infinite tridimensional anion in which silicon or aluminum and oxygen or both alternate, forming an open frame network within which sodium ions and water are arranged. The open frame-work permits the gain and loss of water and ion exchange from the crystal. The stability of the ions permits this without disturbance of the structure. In solutions containing calcium ions, such as the liquid phase of concrete, calcium ions can replace sodium ions, and, accordingly, zeolites in an aggregate can be sources of alkali just as well as the cement. As indicated, the removal of water from a zeolite generally does not cause a change in the volume of the unit cell of the crystal. However, the zeolite laumontite is an exception. This mineral loses about one eighth of its water in the formation of the zeolite leonhardite. This occurs with a change of about 1.5 per cent in the volume of the unit cell. Accordingly, when leonhardite reacts with water to laumontite, it expands about 1.5 per cent. Therefore, aggregates containing this zeolite may be the cause of expansion in concrete in which they are used [*6*]. This is simply a solid-liquid hydration reaction similiar to those illustrated by Eqs 1 and 5.

Clays:

There are three general structural types among the clay minerals, layer structures of two types and one fibrous structure. In certain of these minerals with layer structures, some of the layers are separated by interlayer water. These minerals shrink when they lose water and expand when they regain water. Montmorillonites and mixed-layered clays containing expanding layers like montmorillonite may exist in partially dehydrated form in certain rocks. Clays containing expanding layers have been identified as the expansive components in certain limestones and phyllites, which may be characterized as a metamorphic, mica-rich argillaceous shale [*4*]. These reactions, like those of the zeolites, are simple hydration reactions similiar to those illustrated by Eqs 1 and 5.

In C 295, it is stated "certain materials are known to be reactive with the alkalies in cements. These include . . . ; certain zeolites such as heulandite: and certain constituents of some phyllites." From what has been said above about zeolites and clays, it appears that the zeolite and clay minerals that may cause expansion in concrete do so by reaction with water and not by reaction with alkalies. Montmorillonites are capable of cation exchange with the liquid phase of cement pastes: such exchange may either take up alkalies from the liquid phase or release alkalies to it, depending on the nature of the exchange cations of the montmorillonite. There appears to be a need for additional studies in this field to determine the exact role of the alkalies in the behaviors of zeolites and clays.

Dolomitic Limestones:

There have been a number of reports [*2*] of expansions in concrete in which the aggregate was dolomitic limestone. Tests of these rocks showed that the rates at which they produced expansions in concrete increased as the alkali content of the cement increased. Some of these rocks contained considerable amounts of clay and possibly some opaline silica. In these cases there is the possibility that the deleterious reaction is an alkali-silica reaction. However, Hadley [*17*] observed that a specimen of a single crystal of dolomite expanded during immersion in a solution of sodium hydroxide. Also, it was observed that when samples of these rocks that produced expansion in concrete were immersed for some period of time in relatively dilute solutions of alkali hydroxide alkali carbonate appeared in the solution and the solid phase decreased in dolomite content and increased in calcite content. Also a new solid phase, brucite $Mg(OH)_2$, appeared. These results suggest that the mineral dolomite is the reactive component in some of these dolomitic stones and that it takes part in a dedolomitization reaction as illustrated in Eq 6 in which M is either Na or K.

$$CaMg(CO_3)_2 + 2MOH + \text{water}$$
$$= M_2CO_3 + Mg(OH)_2 + CaCO_3 \quad (6)$$

Some dolomitic rocks develop siliceous reaction rims in concrete, but generally this type of rock does not produce much expansion in concrete. On the other hand, dolomitic rocks that produce significant expansion in mortars or concrete generally do not develop the reaction rims. It appears, therefore, that the dolomitic rocks that undergo some reaction in concrete can be divided into two groups:

1. Rocks which expand in an alkaline environment.
2. Rocks which develop siliceous rims in concrete.

Sherwood and Newlon [*18*] agitated powdered specimens of the minerals, dolomite and calcite, separately and as blends of the two with solutions of either sodium or potassium hydroxide of several concentrations. Examinations of the liquid and solid phases indicated that Eq 6 correctly represents the reaction of dolomite with dilute solutions of either sodium or potassium hydroxide. However, their results also indicated that calcite reacts slowly in these solutions as illustrated in Eq 7.

$$\mathrm{CaCO_3 + 2MOH + water = M_2CO_3 + Ca(OH)_2} \quad (7)$$

On the other hand, the results of Sherwood and Newlon showed that dolomite reacted in concentrated solutions of the hydroxides as illustrated in Eqs 8 and 9.

$$\mathrm{CaMg(CO_3)_2 + 2NaOH + water = CaNa_2(CO_3)_2 \cdot 5H_2O^{*} + Mg(OH)_2} \quad (8)$$

$$\mathrm{5CaMg(CO_3)_2 + 12\ KOH + water = 2Ca_2K_6(CO_3)_5 \cdot 6H_2O^{**} + Ca(OH)_2 + Mg(OH)_2} \quad (9)$$

(* = gaylussite; ** = buetschliite)

Their results also showed that calcite reacted with concentrated solutions of these hydroxides as illustrated in Eqs 10 and 11.

$$\mathrm{2CaCO_3 + 2NaOH + water = CaNa_2(CO_3)_2 \cdot 5H_2O + Ca(OH)_2} \quad (10)$$

$$\mathrm{5CaCO_3 + 6KOH + water + Ca_2K_6(CO_3)_2 \cdot 6H_2O + 3Ca(OH)_2} \quad (11)$$

When the dolomitic limestones that produced expansion in concrete were tested for expansions in solutions of sodium, potassium, and lithium hydroxides, the greatest amount of expansion was obtained in sodium hydroxide, the least in lithium, and an intermediate amount in potassium hydroxide. The difference in the behavior of the hydroxides has been explained on the basis of the solid-liquid reaction mechanism [*19*] by which ionic compounds dissolve in water.

Returning to the rocks that form reaction rims, Mather, Buck, and Luke [*20*] found rims on dolomite-free carbonate aggregates in concrete. Some of these rims could be attributed to alkali-silica reaction.

It seems likely that the rim-forming dolomitic rocks contain one or more of the reactive forms of silica which is responsible for the rims and for the expansions observed with some of these rocks. The dolomitic limestones that produce expansions in concrete differ from the usual carbonate rocks that have given good service as concrete aggregates in being made up of very fine crystals of dolomite and calcite associated with relatively large amounts of clay and other insoluble materials. The texture of the rock as revealed by the microscope, the expansions of specimens of the rock in sodium hydroxide, and the expansions of concrete beams made with cements of high alkali contents appear to be criteria that may be used in the development of methods for evaluating the potential reactivity of these rocks in concrete.

EVALUATION OF TEST METHODS

Much of the work devoted to the development of test methods for predicting the performance of cement-aggregate combinations has been in the field of the alkali-silica reaction as illustrated in Eq 2. ASTM Specifications C 227 and C 289 were developed for use in studies of aggregates in which the principal deleterious reaction was the alkali-silica reaction. In general, there has been fairly good correlation between the results obtained with these methods and the field performance of the aggregates in concrete. Work is now under way to determine whether or not the results obtained with ASTM Specification C 227 will be useful in predicting the performance of concrete containing aggregates that undergo the alkali-carbonate reactions illustrated in Eqs 6–11.

Among the purposes for developing ASTM C 295 was that of identifying materials in aggregates that might be reactive not only with alkalies but also with water and oxygen as illustrated in Eqs 1–11 and with the object of estimating the quantities present. By following the procedures of C 295, experienced petrographers are able to identify most of the reactive materials in aggregates and to obtain an estimate of the quantities present. However, there is need for improvement in this field and it is believed that recommended practices similiar to that of C 295 should be prepared for the use of other procedures, such as, differential thermal analysis and X-ray and electron diffraction. These are now being used in research laboratories for the identification and study of clay and zeolite minerals.

ASTM Method C 342 was the outgrowth of work by Conrow [*21*] in developing a method for predicting the performance of cement-sand-gravel combinations. The particular reaction involved has been called a cement-aggregate reaction because of the feeling that the reaction might not be an alkali-silica reaction. However, usually examinations of the concrete from field structures show that some alkali-silica reaction has occurred in which probably reactive forms of silica and natural glasses are both involved. Since one of the components of the aggregate is an opaline limestone, it may be that the opaline silica in this limestone reacts as illustrated in Eq 2 and that the calcium carbonate reacts as illustrated in one or all of Eqs 7, 10, and 11. Although this report is concerned primarily with chemical reactions, it should be pointed out that some investigators [*7*] have suggested that much of the distress in concrete made with these sand-gravel aggregates may be physical failure resulting from the normal cycles of wetting and drying and temperature changes because of: (*a*) a very low coefficient of thermal expansion, (*b*) poor bond with the cement paste, and (*c*) high rigidity (low compressibility) of the coarse aggregate.

ASTM Method C 342 calls for the use of the aggregate as a fine aggregate, whereas Conrow used the entire sand-gravel aggregate in his specimens. ASTM Subcommittee II-b of Committee C-9, Reactions of Aggregates in Concrete, prepared and distributed for information to the members of C-9 a draft of a method based on the use of the entire sand-gravel aggregate under Tentative Method of Test for Potential Volume Change of Cement-Aggregate Combinations in Concrete. However, since the method is not designed to be useful with aggregates other than those used as sand-gravel aggregates, Subcommittee II-b has not recommended it for adoption by ASTM as a standard method.

There appears to be a need for writing standard test methods for use in evaluating the performance of cement-aggregate combinations in which the aggregate undergoes alkali-carbonate reaction. There is a possibility that C 227 with some modifications may be used for this purpose. C 295 is, of course, as useful with these aggregates as it is with the reactive silica-bearing aggregates.

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Concrete Aggregates

SOUNDNESS AND DELETERIOUS SUBSTANCES

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Good concrete must embody adequate workability, strength, and durability. The most important of these is durability. It is futile to provide ease of placement and an excess of load-carrying capacity if the structure does not remain functional and attractive for a reasonable period.

Many factors contribute to concrete's ability to endure: the quality of materials, the proportions, the mechanics of mixing and placement, and the ambient conditions during construction and throughout the life of the structure. An important element is the quality of the aggregates—their soundness and freedom from deleterious substances.

It is difficult to distinguish sharply between measurements which evaluate the soundness of an aggregate and those which identify deleterious substances. Soundness is the more general term, usually considered to be an attribute of the aggregate as a whole. Attempts to measure that property involve representative samples tested in such a way as to predict over-all performance level. In the case of deleterious substances, attention is focused on the individual particles or contaminants that may be harmful to concrete.

The effects of aggregates or of individual particles in them on the durability of concrete depend, at least in large part, upon their volume stability. The constituents of a good aggregate must be able to resist excessively large or permanent changes in volume when subjected to destructive agencies such as freezing and thawing, heating and cooling, or wetting and drying. The volume changes of unsound particles may cause deterioration of concrete ranging from localized pitting and scaling to extensive cracking and deep-seated disintegration. The former are usually of little structural significance but may be detrimental to the appearance of the structure; the latter may lead to structural failure or deficiency.

So-called deleterious substances are harmful to concrete in several different ways. Excessive amounts of very fine material may increase the mixing water requirement. Organic impurities may interfere with the hardening characteristics of cement. Certain mineral constituents may react harmfully with ingredients of the cement, a subject discussed elsewhere in this publication. Finally, certain particles may be so unstable volumetrically as to require more direct attention than is provided by usual tests for over-all aggregate soundness. The nature of deleterious substances varies greatly from locality to locality with the result that a wide diversity of specification requirements and test methods exist [*1–5*].[2]

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

EFFECTS ON CONCRETE

Unsound aggregates fall into two general categories depending upon the nature of the volume changes which they undergo or induce in the concrete. In one class of particles—friable sandstones, soft limestones, ochers, clay lumps, and so on—the damage is caused by failure of the pieces to maintain their integrity. Their volume change may be considered negative, with the individual particles breaking down into numerous smaller pieces. Depending upon the number present, deterioration may be general but, more often, is evidenced by localized surface pitting or scaling [*4*].

A second and more dangerous class of unsound particles consists of those which expand disruptively or produce disruptive expansive forces in concrete. Examples of this type are certain laminar rocks—principally limestones containing clays—and porous chert. Such materials, when frozen in a saturated condition or, occasionally, when merely exposed to water, may increase in volume with the development of sufficient pressure to cause deep-seated disintegration of the concrete [*4,6*]. Absorbed water in highly saturated porous rocks may be forced from the pores during freezing to produce disruption of the surrounding matrix [*7*].

Individually classified deleterious substances of general concern are organic impurities, clay and clay lumps, soft particles, and lightweight pieces, including coal and lignite. Organic impurities, in some but not all cases, retard the hardening of cement and reduce concrete strength, particularly at early ages [*1,8*]. Finely divided coal or lignite in sufficient quantity also will retard hardening [*9*]. However, in the more common form of small quantities of discrete particles, it has no significant effect on strength but may detract from appearance by producing surface pits [*4*]. Certain types of clay, principally the bentonites, may cause large increases in mixing water requirement accompanied by reduced strength and increased volume changes of the concrete [*10,11*]. However, finely divided material *per se* is generally not harmful in typically occurring amounts, and there is evidence that specifications are over-restrictive in limitations on fines [*10*]. Soft particles must be present in rather large quantity to cause reductions in strength [*4*] but can be significant in smaller quantities where surface abrasion is severe. Loose application of the term "soft" has caused it to be associated with unsoundness, a connection which has no basis in logic or experience. Particles of very low density and shale are generally considered to be unsound [*2,3,12–14*].

SOUNDNESS TESTS

The usual method for measuring overall aggregate soundness is ASTM Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88). The scope of that method states that it simply "furnishes information helpful in judging the soundness of aggregates subject to weathering action," but most specifications for concrete aggregate place specific limits on permissible losses. Proper provision is seldom made for the limited significance of the sulfate test which is recognized in the method by providing "escape clauses" in terms of service record or performance tests of the aggregate. The lack of justification for inflexible limits and the need for flexibility of interpretation will be apparent from subsequent discussion.

The sulfate soundness test consists of alternate immersion of a carefully graded and weighed test sample in a solution of sodium or magnesium sulfate and oven drying it under specified conditions. The accumulation and growth of salt crystals in the pores of the particles is thought to produce disruptive internal forces similar to the action of freezing water. Loss is measured after a specified number of cycles, usually either five or ten, in terms of the amount of the sample that will pass a sieve smaller than the size upon which it was originally retained.

TABLE 1—PROPERTIES OF SODIUM AND MAGNESIUM SULFATES.
(Based on Ref. *16* and *Handbook of Chemistry and Physics*, Chemical Rubber Publishing Co., Cleveland, Ohio, 42nd edition.)

Item	Sodium Sulfate	Magnesium Sulfate
Form of salt at sulfate test drying temperature:		
Formula	Na_2SO_4	$MgSO_4 \cdot H_2O$
Molecular weight	142.05	138.49
Specific gravity	2.698	2.57
Form of salt at 70 F in presence saturated solution:		
Formula	$Na_2SO_4 \cdot 10H_2O$	$MgSO_4 \cdot 7H_2O$
Molecular weight	322.22	246.50
Specific gravity	1.464	1.68
Saturated solution at 70 F:		
Specific gravity	1.158	1.297
Per cent dry form of salt by weight	17.08	30.45
Per cent higher hydrate equivalent by weight	38.74	54.19
Derived constants:		
Volume of higher hydrate formed when unit volume of dry form is hydrated by saturated solution[a]	5.6587	4.1342
Weight of dry form yielded by 1 cc of hydrated salt, g	0.6454	0.9439
Weight of dry form in 1 cc of saturated solution, g	0.1978	0.3949

[a] Including higher hydrate released by saturated solution when water is extracted to hydrate the dry salt.

Details of the mechanism by which the sulfate test disrupts rock particles is open to speculation in which many authors have engaged [*15–19*]. It is probable that a combination of actions is involved, including not only the pressure of crystal growth but also the effects of heating and cooling, wetting and drying, and pressure developed by migration of solution through pores. The relative importance of these disruptive effects almost certainly varies with the structure of the particle undergoing test.

Whatever the specific process may involve, it is agreed that the system of alternate soaking in sulfate solution and oven drying causes salt to accumulate in the pores of the rock. At some number of alternations, the quantity of salt reaches a level which, upon rehydration during the next soaking, can not be accommodated within the pore volume.

Garrity and Kriege [*16*] provided comprehensive data on properties of sulfate solutions and observations of the behavior of the salts as they passed through various stages of crystallization and dehydration. Their calculations of theoretical salt accumulation in pores appear to be in error by failure to allow for surplus solution required during each cycle to fill space not occupied by crystals, but their basic system of analysis is invaluable in visualizing the process.

TABLE 2—HYPOTHETICAL ANALYSIS OF SALT ACCUMULATION IN 100 CC OF PORE SPACE DURING SULFATE SOUNDNESS TEST EXPOSURE.

Item	Cycle No.				
	1	2	3	4	5
SODIUM SULFATE TEST					
A. Weight Na_2SO_4 at beginning of cycle, g	0	19.78	38.35	55.78	72.01
B. Volume Na_2SO_4 at beginning of cycle, cc (= A/2.698)	0	7.33	14.21	20.67	26.69
C. Volume $Na_2SO_4 \cdot 10H_2O$ formed during immersion, cc (= 5.6587B)	0	41.49	80.43	(116.99)[a]	(151.03)[a]
D. Volume remaining for saturated solution, cc	100	58.51	19.57		
E. Weight Na_2SO_4 after drying, g (= 0.6454C + 0.1978D)	19.78	38.35	55.78	...	...
F. Volume Na_2SO_4 for which space available to convert to $Na_2SO_4 \cdot 10H_2O$ (= (100 − B)/4.6587)	...	...	...	17.03	15.74
G. Volume $Na_2SO_4 \cdot 10H_2O$ formed (= 5.6587F)	...	...	...	96.36	89.05
H. Volume Na_2SO_4 remaining unhydrated, cc (= B − F or 100 − G)	...	...	...	3.64	10.95
I. Weight Na_2SO_4 after drying, g (= 0.6454G + 2.698H)	...	...	...	72.01	87.02
MAGNESIUM SULFATE TEST					
A. Weight $MgSO_4 \cdot H_2O$ at beginning of cycle, g	0	39.49	74.37	104.59	129.81
B. Volume $MgSO_4 \cdot H_2O$ at beginning of cycle, cc (= A/2.57)	0	15.37	28.94	40.70	50.51
C. Volume $MgSO_4 \cdot 7H_2O$ formed during immersion, cc (= 4.1342B)	0	63.53	(119.63)[a]	(168.25)[a]	(208.82)[a]
D. Volume remaining for saturated solution, cc	100	36.47			
E. Weight $MgSO_4 \cdot H_2O$ after drying, g (= 0.9439C + 0.3949D)	39.49	74.37	...	...	...
F. Volume $MgSO_4 \cdot H_2O$ for which space available to convert to $MgSO_4 \cdot 7H_2O$ (= (100 − B)/3.1342)	...	...	22.67	18.92	15.79
G. Volume $MgSO_4 \cdot 7H_2O$ formed (= 4.1342F)	...	...	93.73	78.22	65.28
H. Volume $MgSO_4 \cdot H_2O$ remaining unhydrated, cc (= B − F or 100 − G)	...	...	6.27	21.78	34.72
I. Weight $MgSO_4 \cdot H_2O$ after drying, g (= 0.9439G + 2.57H)	...	...	104.59	129.81	150.85

[a] Reimmersion at this stage theoretically yields a volume of the higher hydrate exceeding the available pore space. For this comparison, it is assumed that, thereafter, hydration proceeds only to the extent that the total volume of hydrated and unhydrated salt exactly fills the available pore space.

The schematic comparison of salt accumulation discussed below involves use of constants provided in their paper as well as information secured from the Handbook of Chemistry and Physics and the International Critical Tables.

Table 1 shows characteristics of sodium and magnesium sulfate relevant to their function in the soundness test. Also shown are certain constants, derived from their basic properties, which assist in making the calculations of salt accumulation. Table 2 constructs a purely comparative hypothetical analysis of what may happen as cycles of the sulfate test progress. The reader will recognize

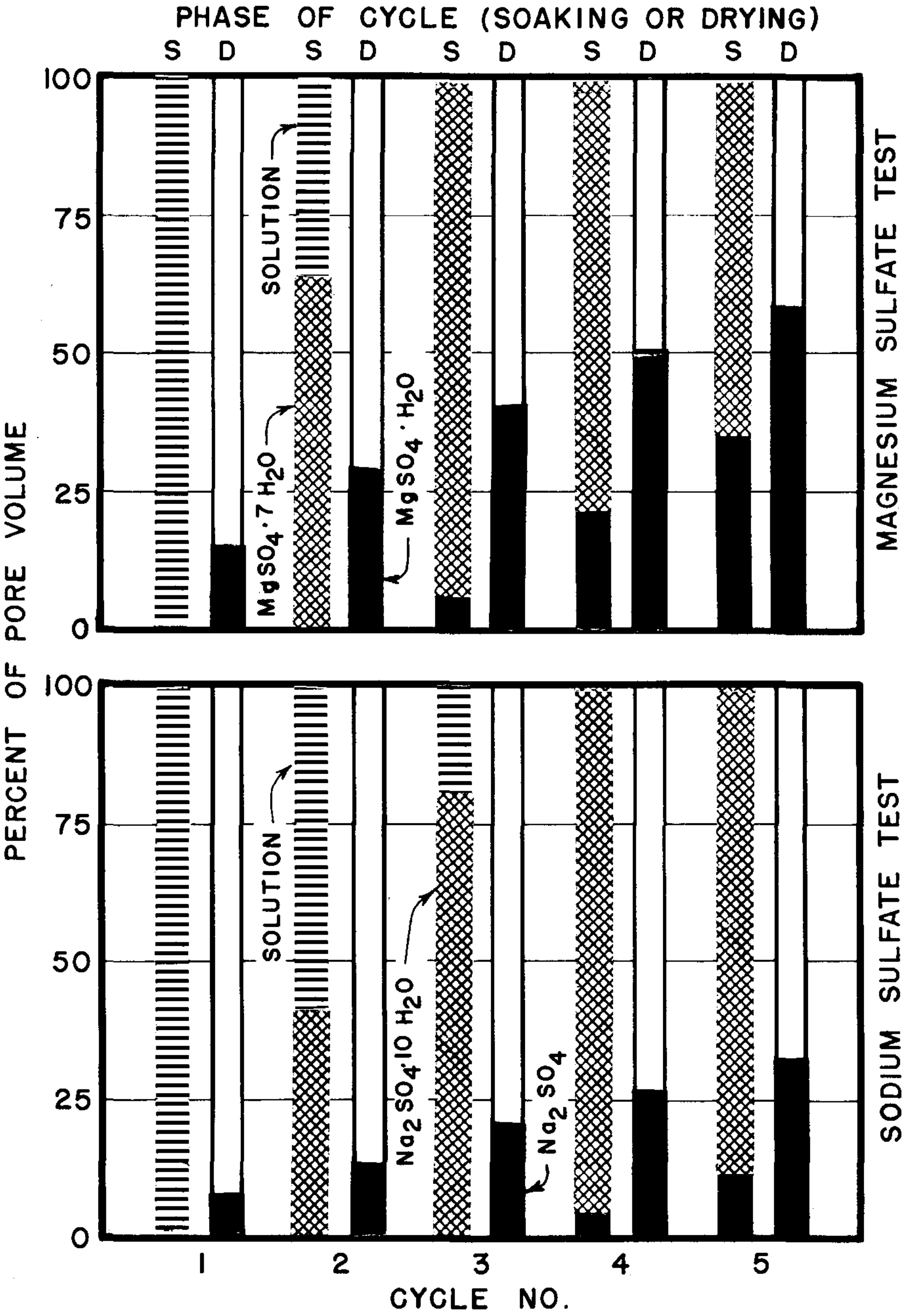

FIG. 1—Salt accumulation in sulfate soundness tests (schematic).

that the process is idealized and that many obstacles would conspire against quantitative accuracy. The data from Table 2 are shown graphically in Fig. 1.

The hypothetical sequence of events for the sodium sulfate test is as follows:

1. After the soaking phase of Cycle 1, the pores are filled with saturated sodium sulfate solution.
2. During drying, the water is driven off and the anhydrous salt, Na_2SO_4, remains.
3. Soaking for Cycle 2 hydrates the Na_2SO_4 to $Na_2SO_4 \cdot 10H_2O$. The water required for this hydration must come from the saturated solution, which releases additional $Na_2SO_4 \cdot 10H_2O$, bringing the total to 41.49 per cent of the pore volume and leaving 58.51 per cent for additional saturated solution.
4. Drying after Cycle 2 leaves the anhydrous salt derived both from the $Na_2SO_4 \cdot 10H_2O$ crystals and from the saturated solution.
5. Cycle 3 follows a similar pattern but increases the residual Na_2SO_4 remaining after drying.
6. During soaking for Cycle 4, insufficient water is available from the saturated solution to hydrate all of the residual Na_2SO_4. Although the table and diagram indicate that some of the Na_2SO_4 remains unhydrated, it seems more probable that crystal development would tend to continue, deriving water from outside the pores. Whatever the exact mechanism or the constitution of the end products, it would appear that disruptive effects of the test should become pronounced at this "critical" cycle when available pore volume is less than the space required for the completely hydrated salt.

Comparison with the similar analysis for the magnesium sulfate test suggests that it should be more severe than sodium. The "critical" stage, at which the hydrated salt theoretically fills all available pore volume while some of the lower hydrate, $MgSO_4 \cdot H_2O$, is still present, occurs during the third cycle rather than the fourth.

INTERPRETATION OF SULFATE SOUNDNESS TESTS

The over-all mechanism by which sulfate tests break up rock particles is not fully understood but, as already stated, probably involves several processes. It has been indicated [*16*,*20*] that crystal growth in a pore should tend to follow the path of least resistance and, therefore, should not exert disruptive pressure until the crystal volume equals the pore volume. At that stage, corresponding to the critical cycle mentioned above, further crystal growth could produce pressure [*20*].

There is evidence that other destructive forces must be involved in the sulfate test exposure. Wuerpel [*18*] found loss to be approximately proportional to number of cycles, casting some doubt on the "critical" cycle idea which presumes a sudden increase in severity after a specific cycle. Appreciable losses have been found to occur when the test is run with distilled water instead of sulfate solution, pointing to the contribution of simple wetting and drying or heating and cooling to disruption of particles [*16*]. Finally, severity of the test has been shown to increase when drying time is extended beyond that needed to dehydrate the crystals [*16*], suggesting damage from the prolonged heating *per se*.

Since the usual purpose of the sulfate test is to evaluate resistance of aggregate to the destructive effects of freezing and thawing, it is appropriate to examine the degree to which a correlation should be expected. The tendency of a rock particle to cause damage of concrete during freezing depends upon its physical characteristics—size, shape and quantity of pores, elastic properties, and external dimensions [*6,7*]. Naturally the performance of concrete will also involve many other factors ranging from the properties of the other ingredients and their proportions to the severity of the exposure.

There is little or no support, either theoretical or experimental, for the assumption that the sulfate test simulates exposure to freezing and thawing in concrete or provides a reliable indication of field performance. The growth of sulfate crystals in pores is not analogous to the development of pressure by an advancing front of freezing water. According to Verbeck and Landgren [*7*], "the mechanism of disruption (in sulfate soundness) is different" and "such test results have only rough and uninterpretable empirical correlation with concrete performance." Reason and research suggest that sulfate soundness test loss should depend more upon the quantity of pores than upon their more critical characteristics of size and distribution [*4,21–25*]. To the extent that the sulfate test correlates with the amount of pore volume and to the extent that this property relates to concrete durability, the test might be assumed to have significance for its intended purpose. However, it would seem more logical to measure pore volume directly by a simple absorption test rather than by the more complicated sulfate test. Data in the literature show as good or better correlations between aggregate absorption and resistance of concrete to laboratory freezing and thawing than between sulfate soundness and freezing and thawing [*6,25*].

In spite of the lack of quantitative significance of sulfate tests in terms of field performance [*1,15,21,22*], specifying agencies persist in putting inflexible limits on the soundness loss and disregarding the need for engineering judgment in its interpretation. These arbitrary limits cause rejection of highly acceptable aggregates and permit acceptance of materials that contribute to extensive concrete disintegration. Nevertheless, recent years have seen an encouraging trend toward more reliance on actual performance in concrete, either in the field or in laboratory freezing and thawing tests, with sulfate tests being relied upon principally when data from more reliable sources are not available.

It has been suggested that the sulfate soundness test might be used to accept aggregates but not reject them [*26*], the assumption being that aggregates which will satisfactorily withstand the test are good while those that break down may or may not be bad. Unfortunately, the test is not reliable even to that extent. Certain aggregates with extremely fine pore structure show almost no loss in the sulfate test but disrupt concrete with explosive violence when frozen in a saturated condition. Conversely, certain highly porous aggregates disintegrate readily in the sulfate test but produce concretes of high resistance to freezing and thawing. A low soundness test loss is usually, but not always, evidence of good durability, whereas a high loss places the aggregate in a questionable category until performance data become available.

REPRODUCIBILITY OF SULFATE SOUNDNESS TESTS

Subsequent to its issuance in 1931, ASTM Method C 88 went through a dozen revisions as tentative before being advanced to a standard in 1963. The revisions were aimed at improving within-laboratory and laboratory-to-laboratory reproducibility, an objective which has been only partially realized. Researches [*4,16–18,27*] have shown that all of the following influence the magnitude of loss measured:

1. Amount of salt in solution, as affected by method of preparation and control of temperature.
2. Purity of the salt.
3. Efficiency of drying oven.
4. Length of drying time.
5. Type of sample container, particularly as it affects ease of drying.
6. Technique of sieving in the preparation of samples and measurement of loss.

Confusion has been further compounded by the multiplicity of alternative procedures recognized within the method. Either sodium or magnesium sulfate may be used; the number of cycles is not designated, but both five and ten are widely specified; and, until recently, different sieve size distributions of coarse aggregate test samples have been permissive. For any given aggregate, the magnitude of loss will depend upon which combination of these variables is designated by the specifying agency.

Woolf [*26,28*] and others [*19,27*] have long advocated reforms to reduce the number of permissible combinations of test procedure as well as to reduce variations attributable to differences in apparatus and technique within the method. That crusade yielded the following worthwhile changes which were incorporated during the few years preceding advancement to standard:

1. Inclusion of limitations on oven efficiency to assure satisfactory drying capability.
2. Elimination of alternate gradings for coarse aggregate samples.
3. Provision for final sieving of coarse aggregate over sieves with openings five sixths as large as those used in preparing the sample.

The latter was intended to reduce variations traceable to performing the final sieving over the original retaining sieve. That practice often permitted particles to be classed as loss which were barely retained in preparing the sample but slipped through on final sieving because of slight attrition or more vigorous manipulation. Obviously, changing to the five-sixth ratio of sieve opening reduces the measured loss, although the difference is probably of little consequence in relation to the limited over-all significance of the test.

Other recommendations for simplification of sulfate soundness testing have not fared well. It has been proposed that the test be performed on a single size of either coarse or fine aggregate. This would reduce the amount of testing and do away with calculation of weighted losses based on grading. The objection has been that no one size adequately represents the soundness of a graded aggregate. Particularly in the case of heterogeneous gravels, there may be large differences in soundness between the various size fractions. Similarly, the proposal that one or the other sulfate —either sodium or magnesium—be designated exclusively has been strenuously opposed. Proponents of the idea have generally favored magnesium sulfate on the basis that its solubility is less sensitive to temperature changes than is sodium [*19,27*]. Opponents point to the dislocation that would be caused by switching to a different salt than the one upon which the experience used to establish specification limits has been based.

Except for the possible advantage of magnesium sulfate in terms of control of test solutions, there seems to be no superiority of any one version of the sulfate test over the others. General relationships developed for Subcommittee III-e of Committee C-9 [*29*] have shown trends relating the various procedures. Although these are not quantitative in the sense of permitting translation of results for one type of salt or number of cycles to another, they do indicate approximate degree of severity in a statistical sense. In the case of fine aggregate, magnesium sulfate is consistently more severe than sodium, producing losses averaging about twice as great; for coarse aggregate, magnesium is again the more severe on the average, but the difference is smaller and, for many individual aggregates, may be reversed. Losses at ten cycles average about 50 per cent higher than at five cycles for either type of salt and for both coarse and fine aggregate. Loss through the half size sieve averages 60 to 70 per cent of the loss through the full size original retaining sieve. (Note that the recent revision of ASTM Method C 88 changed from the full size to the five-sixth size sieve for final sieving. The resultant reduction in measured loss should be nominal in view of the demonstrated modest difference between full- and half-size sieve criteria.)

Early data [*16*] secured before test procedures were closely controlled, indicated very poor reproducibility of sulfate tests on the same material within laboratories as well as from laboratory to laboratory [*16,17*]. It appears that refinements during the intervening years have improved this situation. Unpublished detailed data on 5-cycle sodium and magnesium sulfate tests made in triplicate on 36 fine aggregates and 56 coarse aggregates provide the following within-laboratory-single-operator statistics [*30*]:

Type of Test	Standard Deviation (of numerical loss)	Coefficient of Variation, per cent
Coarse aggregate:[a]		
5-cycle magnesium sulfate	1.1	14.5
5-cycle sodium sulfate	1.1	12.4
Fine aggregate:[b]		
5-cycle magnesium sulfate	0.4	3.9
5-cycle sodium sulfate	0.3	6.0

[a] Fixed grading, 1-in. max size.

[b] Typical concrete gradings; loss weighted in accordance with individual grading.

To the extent that these data apply generally, which presupposes meticulous attention to all details of testing, duplicate determinations by one operator within one laboratory should be expected 95 per cent of the time to yield numerical losses checking within about 4 for coarse aggregate and 1½ for fine aggregate.

Available data and informal comparisons sponsored by Subcommittee III-e of ASTM Committee C-9 discourage any attempt to estimate laboratory-to-laboratory reproducibility of sulfate soundness tests. Failure in many cases to secure results of even comparable magnitude probably stems from two causes: differences in severity producible within the prescribed limits of the standard method (such as the demonstrated damage from extra drying beyond attainment of essentially constant weight); and active deviations from the prescribed method (such as shortening the drying period to achieve a convenient time schedule or using nonstandard containers). The experiences suggest that a further precaution might be needed in the scope of Method C 88: Not only is the test limited to a very rough indication of soundness but should be confined to comparisons between aggregates where the tests are made by identical techniques, preferably simultaneously.

OTHER TESTS FOR SOUNDNESS

Freezing and thawing tests of unconfined aggregate have been the most frequently suggested alternative to sulfate soundness [*18,31–34*]. The method would appear to have the merit of producing a destructive action more directly comparable to that of severe exposure of concrete. However, Verbeck [*7*] has pointed out that the destructive effects of particles freezing in concrete are not simulated by freezing them in an unconfined state. Therefore, the unconfined tests provide little more useful information than the sulfate tests. Various investigators have reported general correlations between aggregate freezing and thawing and other tests including absorption and sulfate soundness, and in some cases have expressed confidence in their ability to predict field performance.

Freezing and thawing of unconfined aggregate in a dilute solution of alcohol is the most recently suggested rapid method for evaluating soundness [*34*]. Limited experience suggests that the test may be indicative of field service for certain classes of aggregate. The use of alcohol accelerates the destructiveness and permits the test to be completed in about the same time as sulfate soundness.

There is evidence that standardized freezing and thawing tests of concrete provide the best information on the soundness of aggregates. Considerable data have been developed showing good correlation between field performance of aggregates and the resistance to freezing and thawing in the laboratory of concretes made with them [*22,35–40*]. As yet, the laboratory methods have not been refined to the point where specification limits can be established or the test results interpreted quantitatively with respect to aggregate soundness. This can be attributed to the large number of variables other than aggregate which affect the results, including properties of the other materials, air entrainment, water-cement ratio, age of the concrete at the time of test, moisture condition of the aggregate and concrete, and the nature of the freezing and thawing exposure.

Encouraging efforts have been made in applying new concepts of the significance of concrete freezing and thawing to the evaluation of aggregate performance. Tremper and Spellman [*40*] have proposed that aggregates be compared on the basis of the length of time concrete made with them will resist becoming saturated to the point of being vulnerable to freezing and thawing. That approach might also permit the aggregate selection to be weighed in relation to the severity of field exposure anticipated.

The general subject of concrete freezing and thawing is discussed elsewhere in this publication.

DELETERIOUS SUBSTANCES

The variety of materials potentially harmful to concrete is so extensive as to defy enumeration. Contaminants may be introduced from many sources, including the aggregates, and their manifestations will vary depending upon many factors involved with production, treatment, and exposure of the concrete. The discussion here is limited to materials in aggregates which are recognized as deleterious and occur with sufficient frequency to require some sort of regulation.

Several investigators have attempted to classify deleterious substances. Swenson and Chaly [*5*] have drawn on earlier literature [*1,4*] to establish a classification based on "external" and "internal" properties, both chemical and physical. Potentially objectional physical features in the external category are encrustations, highly weathered surfaces, highly polished surfaces, undesirable shape, and extreme fineness. Internal properties of concern are undesirable pore characteristics, high-volume changes during wetting and drying, laminations, lack of strength, and unfavorable thermal characteristics.

Many deleterious particles or undesirable characteristics of particles can be recognized only by petrographic examination—a subject discussed elsewhere in this publication. Specific standardized tests are available for a few types subject to limitations in the ASTM Specification for Concrete Aggregates (C 33), as discussed below.

Clay Lumps:

Many specifications, including ASTM Specification C 33, place rigorous limitations on the permissible percentage of clay lumps in aggregate. The harmfulness of such particles depends upon their surviving as lumps in the hardened concrete where they may break down from freezing and thawing or wetting and drying to produce unsightly surface pits or popouts. For this reason, the specification limits are more rigorous than they are for the over-all content of clay and silt of which the lumps are composed. In other words, the specification limits for clay lumps are intended to apply only to those agglomerations which are sufficiently sturdy to withstand the concrete mixing operation. The residue from highly friable particles, which break down during mixing, should be curtailed by the specification limits on minus No. 200 material.

In recognition of the important distinction between harmful lumps and particles which merely contribute fines to the concrete, the test for clay lumps, ASTM Test for Clay Lumps in Natural Aggregates (C 142 – 64 T) was recently revised. The test is to be performed after the sample has been tested for minus No. 200 material. The presumption is that particles which disintegrate during the washing operation of the latter test are not capable of surviving as lumps during concrete mixing. As now written, therefore, ASTM Method C 142 is intended to measure only those particles which might cause unsightly blemishes in concrete surfaces.

Lightweight Pieces:

The ASTM Test for Lightweight Pieces in Aggregate (C 123) may be used to determine coal and lignite or other particles for which harmfulness is associated with low specific gravity. ASTM Specification C 33 places limits on the former and also distinguishes potentially harmful chert on the basis of its specific gravity. Coal and lignite are separated at a specific gravity of 2.0. The quantity is limited to 1.0 per cent unless the aggregate is to be used in concrete where surface appearance is critical when the limit is reduced to 0.5 per cent. Potentially harmful chert may be distinguished on the basis of a specific gravity less than 2.35 with the permissible percentage dependent upon the expected severity of exposure.

Coal and lignite in the form of sizable particles may cause localized pitting and staining of concrete surfaces. Determination that a particle is lighter than 2.0 in specific gravity does not, in itself, convict it of being coal or lignite. This fact, as well as the knowledge that lightweight particles of other types are less detrimental to appearance, led to a recent revision of ASTM Specification C 33 to provide that "only material (less than 2.0 specific gravity) that is brownish-black or black shall be considered coal or lignite." It is recognized that even this criterion is not infallible and that petrographic examination may be necessary for positive identification. Very finely divided coal or lignite in appreciable quantity may cause strength reduction of concrete [*9*], but this effect will be detected by the test for organic impurities discussed below.

The use of the test for lightweight pieces to identify potentially harmful chert applies only to coarse aggregate, presumably because particles must be larger than a "critical size" to produce disruptively expansive forces in concrete [*7*]. The vulnerability of the chert particle depends upon its pore structure which, in turn, has been found to relate approximately to specific gravity. The significance of pore structure and its relationship to freezing and thawing resistance are discussed elsewhere.

The test for lightweight pieces is probably repeatable within about 10 per cent of the measured content (95 per cent probability level) on duplicate portions of the same sample [*14*].

Clay and Silt:

ASTM Test for Materials Finer than No. 200 Sieve in Mineral Aggregates by Washing (C 117 – 62 T) provides a measure of fines, including clay and silt, in concrete aggregates. It has been criticized as not furnishing an indication of harmful clays which may increase mixing water requirements and volume change tendencies of concrete [*11*]. ASTM Specification C 33 recognizes that rock dust is less detrimental than clay by increasing the limits when the fines consist "of the dust of fracture, essentially free from clay or shale."

Tests have been developed which are reported to indicate reliably the potential harmfulness of clay constituents in concrete aggregates [*11*]. These tests—sand equivalent and sedimentation—evaluate the clay in terms of the extent to which a water suspension prepared from the aggregate will settle in a prescribed period. The tests are reported to correlate well with mixing water requirement of concrete which, in turn, affects shrinkage and strength. It might be conjectured that, in view of the many other factors which affect mixing water requirements, aggregates might best be evaluated for this property by actual direct comparisons in concrete.

Experiments to establish the precision of the test for minus No. 200 material, ASTM Method C 117 [*41*], have indicated that an individual operator should have about a 95 per cent probability of checking his result within 0.3 in duplicate determinations on the same sample.

Organic Impurities:

Certain types of organic matter—principally tannic acid and its compounds derived from the decay of vegetable matter—interfere with the hardening and strength development of cement [*8,9*]. The colorimetric test for organic matter, ASTM Test for Organic Impurities in Sands for Concrete (C 40), detects such materials but unfortunately also reacts to other organics—bits of wood, and so on—which are not significantly harmful to strength. A negative test, therefore, is conclusive evidence of freedom from harmful organic matter, but a positive reaction (dark color) may or may not forebode difficulty.

The colorimetric test is employed as a warning of possible danger from organic matter, to be checked by mortar strength tests. Until its recent revision, the long-standing version of the mortar test, ASTM Test for Measuring Mortar-Making Properties of Fine Aggregate (C 87 – 62 T) was criticized for its inability to evaluate organic impurities independently of other sand properties which affect strength. That method compared the suspect sand with Ottawa sand of a standard grading in mortars of fixed water-cement ratio. The strength ratio, which was intended to indicate whether or not the organic matter was harmful, was actually more affected by the grading, shape, and texture of the sand [*42*]. The recent revision, ASTM Test for Effect of Organic Impurities in Fine Aggregate on Strength of Mortar (C 87 – 63 T), attempts to remove this difficulty by using as the basis of comparison the suspect sand treated to remove the organic matter. Presumably the only difference between the test mortar and reference mortar is the presence of organic matter in the former.

The colorimetric test yields a highly reliable and reproducible indication of whether or not organic matter is present in sand. The check for effects on mortar strength by ASTM Method C 87 is subject to the problems of strength testing in general, discussed elsewhere. ASTM Specification C 33 allows the mortar strength ratio to be 95 per cent, the purpose of the slight reduction being to provide for unavoidable testing variation.

Soft Particles:

The property of softness in individual particles would appear to be significant only in relation to wear resistance or, possibly, strength of concrete. By general usage, however, the term "soft particle" has become almost synonymous with "unsound particle." Not infrequently, so-called soft particles are determined by visual examination on the basis of experience and include rock which may or may not actually be soft but is considered likely to produce popouts, pits, or stains. This looseness of terminology has led to confusion and misunderstanding in the establishment of limits on particles which are actually soft but not necessarily harmful in the other respects mentioned.

Even large percentages of soft particles in aggregates have been shown to detract relatively little from the strength of concrete [*4*]. Furthermore, strength effects of aggregate are best regulated by limits on the concrete itself or may be evaluated in a very general way by other tests such as Los Angeles abrasion.

The test by which soft particles are measured, ASTM Test for Scratch Hardness of Coarse Aggregate Particles (C 235 – 62 T), is not intended to evaluate any other property. It is a simple determination of whether a brass stylus of designated hardness will scratch the particle. It, therefore, provides an arbitrary dividing line on one side of which particles are classed as "soft." There is every reason to suppose that the quantity of such particles might be related to the performance of concrete subject to severe abrasion, such as in a heavy duty floor exposed to the action of steel wheels or rubber-tired rollers. Except where abrasion resistance is critical, however, specific limitation of soft particles as measured by ASTM Method C 235 cannot be justified.

CONCLUSION

Sulfate soundness tests provide only a very rough indication of the ability of concrete aggregates to produce durable concrete. If properly interpreted in the light of other tests and field service record, they assist engineering judgment in determining the permissibility of using a particular aggregate in a particular application. A specific limit on sulfate test loss to distinguish between acceptable and unacceptable aggregate is not justified. If the limit is set low enough to reject all objectionable aggregates, it will also reject most acceptable ones. If it is high enough to accept all good aggregates, it will also accept many that should not be used.

Other tests for soundness appear not to be appreciably more definitive. Service record is the engineer's best criterion for predicting aggregate performance. The next best source of information is comparison of aggregates in laboratory freezing and thawing tests of concrete. Even these tests must be interpreted in relation to the expected severity of exposure and to economic availability. They serve primarily to rank aggregates in order of probable relative endurance rather than to provide a quantitative measure of life expectancy in the field.

Tests for deleterious particles in aggregates are highly useful if the results are realistically applied in relation to the expected exposure of the concrete. Restrictions on clay lumps are imposed for the purpose of minimizing surface blemishes; they should be applied only to particles that survive the concrete mixing operation and persist as lumps after the concrete is in place.

Lightweight pieces can be accurately determined by ASTM Method C 123. The level selected for specific gravity separation, as well as the numerical limits, should be such as to restrict particles whose low density is related to objectionable behavior in concrete—such as, development of pits, stains, or popouts.

Excessive fines as determined by ASTM Method C 117 are objectionable if they increase the mixing water requirement of concrete or contain clays which aggravate shrinkage. The test does not distinguish clay from less harmful material, such as fine sand or rock dust, a fact which should be recognized in setting specification limits.

Organic impurities are readily detected by ASTM Method C 40. Their potential harmfulness to strength development of cement must be checked by ASTM Method C 87.

ASTM Scratch Hardness Test (C 235) provides an arbitrary basis for classifying particles as soft. The quantity of such particles is of significance and should be specified only where surface abrasion resistance of the concrete is critical.

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PART IV

Tests and Properties of Other Materials

STP169A-EB/Apr. 1966

Other Materials

MIXING AND CURING WATER FOR CONCRETE

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This paper is primarily concerned with the significance of tests of various types of waters for mixing and curing concrete and makes no attempt to include the effect of quantity of mixing water. Almost all natural waters, fresh waters, and waters treated for municipal use are satisfactory as mixing water for concrete if they have no pronounced odor or taste. Because of this very little attention is usually given to the water used in concrete, a practice that is in contrast to the frequent checking of the cement and aggregate components of the concrete mix.

MIXING WATER

A popular criterion as to the suitability of water for mixing concrete is the classical expression, "If water is fit to drink it is all right for making concrete." This does not appear to be the best basis for evaluation, since some waters containing small amounts of sugars or citrate flavoring would be suitable for drinking but not mixing concrete [*1*],[2] and, conversely, water suitable for making concrete may not necessarily be fit for drinking. In an attempt to be more realistic some concrete specification writers attempt to insure that water used in making concrete is suitable by requiring that it be clean and free from deleterious materials. Some specifications require that if the water is not obtained from a source that has proved satisfactory, the strength of concrete or mortar made with the questionable water should be compared with similar concrete or mortar made with water known to be suitable. The Corps of Engineers, U.S. Army Specifications [*2*], in addition to a general description of acceptable water also states that if the pH of water lies between 6.0 and 8.0 and the water is free from organic matter, it may be regarded as safe for use in mixing concrete. (An exception to this is the case where sodium sulfates or other natural salts are present in excessive amounts.) These specifications also state that if water is of questionable quality it should be tested in mortar cubes for which the 7 and 28 day compressive strengths should equal at least 90 per cent of that of companion test specimens in which distilled water is used before it is judged to be acceptable [*3*]. Other than comparative tests of this type, no special test has been developed for determining the quality of mixing water, and, hence, it is difficult for the man in the field to judge the fitness of water for use in concrete [*4*].

The two principal questions regarding mixing water appear to be how do impurities in the water affect the concrete and what degree of impurity is permissible? The following discussion is a resumé of available information on these two items.

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

Effect of Impurities in Mixing Water:

The most extensive series of tests on this subject was conducted by Abrams [*5*]. Approximately 6000 mortar and concrete specimens representing 68 different water samples were tested in this investigation. Among the waters tested were sea and alkali waters, bog waters, mine and mineral waters, and waters containing sewage and solutions of salt. Tests with fresh waters and distilled water were included for comparative purposes. Setting time tests on cement and concrete strength tests from 3 days to 2.33 years were conducted for each of the various water samples. Some of the more significant conclusions based on these data are as follows:

1. The time of setting of portland cement mixes containing impure mixing waters was about the same as those observed with the use of clean fresh waters with only a few exceptions. In most instances the waters giving low relative compressive strength of concrete caused slow setting, but, generally speaking, the tests showed that time of setting is not a satisfactory test for suitability of a water for mixing concrete.

2. None of the waters caused unsoundness of the neat portland cement pat when tested over boiling water.

3. In spite of the wide variation in the origin and type of waters used, most of the samples gave good results in concrete due to the fact that the quantities of injurious impurities present were quite small.

4. The quality of mixing water is best measured by the ratio of the 28 day concrete or mortar strength to that of similar mixes with pure water. Waters giving strength-ratios which are below 85 per cent in general should be considered unsatisfactory.

5. Neither odor nor color is an indication of quality of water for mixing concrete. Waters which were most unpromising in appearance gave good results. Distilled waters gave concrete strengths essentially the same as other fresh waters.

6. Based on a minimum strength-ratio of 85 per cent as compared to that observed with pure water, the following samples were found to be unsuitable for mixing concrete: acid water, lime soak water from tannery waste, carbonated mineral water discharge from galvanizing plants, water containing over 3 per cent of sodium chloride or 3.5 per cent of sulfates, and water containing sugar or similar compounds. The concentration of total dissolved solids in these waters was over 6000 ppm except for the highly carbonated water which contained 2140 ppm total solids. These data support one of the principal reasons for the general suitability of drinking water, for few municipal waters contain as much as 2000 ppm of dissolved solids and most contain far less than 1000 ppm. For drinking waters, 1000 ppm is the maximum permissible in some specifications [*6*]. Very few natural waters other than sea water contain more than 5000 ppm of dissolved solids [*7,8*].

7. Based on the minimum strength-ratio of 85 per cent, the following waters were found to be suitable for mixing concrete:

(*a*) bog and marsh water;

(*b*) waters with a maximum concentration of 1 per cent SO_4;

(*c*) sea water, but not for reinforced concrete;

(*d*) alkali water with a maximum of 0.15 per cent Na_2SO_4 or NaCl;

(*e*) pumpage water from coal and gypsum mines; and

(*f*) waste water from slaughterhouses, breweries, gas plants, and paint and soap factories.

Many of the specifications for water for mixing concrete, especially those which require that it be potable, would have excluded nearly all of the above waters, but contrary to this rather general opinion, the test data show that the use of many of the polluted types of water did not result in any appreciable detrimental effect to the concrete. The important question is not whether impurities are present, but do impurities occur in injurious quantities? It should be noted that the conclusions in Paragraph 7 are based entirely on tests of specific samples from the indicated sources, and it should not be assumed that all waters of the type described would be innocuous when used as mixing water.

Typical analyses of natural fresh water as reported by the U. S. Geological Survey are given in Table 1. Collins [9] reports that these analyses represent public water supplies used by about 45 per cent of the cities of the United States that have a population of more than 20,000.

TABLE 1—TYPICAL ANALYSES OF CITY WATER SUPPLIES (PPM).[a]

Analysis Number	1	3	5	6	7
Silica (SiO_2)	2.4	12.0	10.0	9.4	22.0
Iron (Fe)	0.14	0.02	0.09	0.2	0.08
Calcium (Ca)	5.8	36.0	92.0	96.0	3.0
Magnesium (Mg)	1.4	8.1	34.0	27.0	2.4
Sodium (Na)	1.7	6.5	8.2	183.0	215.0
Potassium (K)	0.7	1.2	1.4	18.0	9.8
Bicarbonate (HCO_3)	14.0	119.0	339.0	334.0	549.0
Sulfate (SO_4)	9.7	22.0	84.0	121.0	11.0
Chloride (Cl)	2.0	13.0	9.6	280.0	22.0
Nitrate (NO_3)	0.54	0.1	13.0	0.2	0.52
Total dissolved solids	31.0	165.0	434.0	983.0	564.0

[a] Taken from Collins [9].

TABLE 2—TOLERABLE CONCENTRATIONS OF IMPURITIES IN MIXING WATER.

Impurity	Maximum Tolerable Concentration
1. Sodium and potassium carbonates and bicarbonates	1 000 ppm
2. Sodium chloride	20 000 ppm
3. Sodium sulfate	10 000 ppm
4. Calcium and magnesium bicarbonates	400 ppm of bicarbonate ion
5. Calcium chloride	2% by weight of cement in plain concrete
6. Iron salts	40 000 ppm
7. Sodium iodate, phosphate arsenate and borate	500 ppm
8. Sodium sulfide	even 100 ppm warrants testing
9. Hydrochloric and sulfuric acids	10 000 ppm
10. Sodium hydroxide	0.5% by weight of cement if set not affected
11. Salt and suspended particles	2 000 ppm

A concrete manual [*10*] published in Denmark in 1944 points out that humic acid and other organic acids should be avoided because their presence means a danger to the stability of concrete.

An article appearing in a 1947 issue of the British publication *Concrete and Constructional Engineering* [*11*] discusses the harmful effects of using acid waters in concrete and claims that the harmful effects of organic acid are not evident as soon as those of mineral acids, while deleterious salts have a greater effect on strength at an early age than at later ages.

Kleinlogel [*12*] in his book *Influences* *on Concrete* states that mixing water should not contain humus, peat fiber, coal particles, sulfur, or industrial wastes containing fat or acid.

A recent article in the *Indian Concrete Journal* [*13*] contains a tabulation of maximum limits for impurities in mixing water which is summarized in Table 2.

The limits for suspended particles in Table 2 agree with the requirements of the U.S. Bureau of Reclamation [*14*] which has a turbidity limit of 2000 ppm for mixing water.

Sugar is probably the organic contaminant that causes the most concern. Steinour [*15*] explains that, although sugar as a contaminant in the field has gained a bad reputation as a retarder and strength reducer, these judgments need qualification. Laboratory tests have shown that, although the smaller amounts retard the setting, they increase the strength development. With larger amounts the setting is further retarded, and early strengths such as those for two and three days (or even seven days) are severely reduced. The later strengths, however, are increased, or at least not adversely affected, provided proper curing is maintained as required. With still larger amounts the cement becomes quick setting and strengths are markedly reduced for 28 days and probably permanently. The amount of sugar that can cause these different effects varies with the other factors involved, such as the composition of the cement, cement content, and ambient conditions.

Use of Sea Water in Mixing Concrete:

In addition to the supporting reference previously mentioned in Abrams' paper [*5*], the English article [*11*] also states that sea water with a maximum concentration of salts of the order of 3.5 per cent does not appreciably reduce the strength of concrete although it may lead to corrosion of reinforcement.

A paper by Liebs in the German publication *Bautechnik* [*16*] contains the results of comparative 7, 28, and 90 day compressive strength tests of concrete mixed with fresh water and with sea water. The data show that the sea water concrete had about 6 to 8 per cent lower strength than the fresh water concrete. No efflorescence was observed. The *Indian Concrete Journal* article [*13*] pointed out that concrete made with sea water may have higher early strength than normal concrete, but strengths at later ages (after 28 days) may be lower. Steinour [*15*] states the use of sea water may cause a moderate reduction in ultimate strength, an effect that can be avoided by the use of a higher cement content. He also notes that concrete in which sea water is used as mixing water is sound but its use may cause efflorescence or dampness, and, in reinforced concrete, the risk of corrosion of the steel is increased. Sea water definitely should not be used for making prestressed concrete.

Hadley [*17*] points out that the sea water was used in the concrete mix for the foundation of the lighthouse at the extremity of the Los Angeles breakwater which was built by the U.S. Engineer Corps in 1910 and that 25 years later it was examined and found to be in good condition with sharp edged corners and no disintegration. There are several references in the literature which indicate that salt water has been used in mixing plain concrete without incurring trouble at later periods. Much of the concrete in the Florida East Coast Railway was mixed with sea water with no detrimental effect due to its use [*18*]. Most engineers are of the opinion that sea water should not be used for mixing reinforced concrete; however, Dempsey [*19*] describes construction of military bases in Bermuda using coral aggregate and concludes that sea water seems to be satisfactory for making reinforced concrete and causes no problem beyond an acceleration in stiffening of the mix. No harmful effect on the durability of reinforced concrete had occurred at the end of four years.

Effect of Algae in Mixing Water on Air Content and Strength of Concrete:

A rather extensive series of laboratory tests reported by Doell [*20*] has shown that the use of water containing algae had the unusual effect of entraining considerable quantities of air in concrete mixes with an accompanying decrease in strength. The data in Table 3 were extracted from Doell's paper and are based on tests with $\frac{3}{4}$-in.-max size aggregate concrete having a water-cement ratio of 0.5 and a slump of 1.5 to 3 in., with the ratio of coarse to fine aggregate constant.

TABLE 3—EFFECT OF ALGAE IN MIXING WATER ON AIR CONTENT AND STRENGTH OF CONCRETE.

Mix Number	Algae in Mix Water, per cent	Air in Concrete, per cent	Compressive Strength, 28 day, psi
10........	none (control)	2.2	4830
8........	0.03	2.6	4840
7........	0.09	6.0	4040
5........	0.15	7.9	3320
9........	0.23	10.6	2470

In addition to the detrimental effect on strength, one of the important aspects of these data is that considerable quantities of air can be entrained in concrete by the use of mix water containing algae.

Effect of Hardness of Mixing Water on Air Content of Concrete:

Wuerpel [*21*] reports data on a series of air determination tests with waters of various degrees of hardness which show that the air content was not affected by the amount of hardness in the water.

Curing Water

There are two primary considerations with regard to the suitability of water for curing concrete. One is the possibility that it might contain impurities that would cause staining and the other is that it might contain aggressive impurities that would be capable of attacking or causing deterioration of the concrete. The latter possibility is unlikely, especially if water satisfactory for use in mixing concrete is employed. In some instances the staining or discoloration of the surface of concrete from curing water would not be objectionable. The most common cause of staining is usually a relatively high concentration of iron or organic matter in the water; however, relatively low concentrations of these impurities may cause staining, especially if the concrete is subjected to prolonged wetting by runoff of curing water from other portions of the structure [*2*].

Test data included in an unpublished Waterways Experiment Station Miscellaneous Paper by the Corps of Engineers, U.S. Army [*22*], show that there is not a consistent relation between dissolved iron content and degree of staining. In some cases 0.08 ppm of iron resulted in only a slight discoloration, and, in other cases, waters with 0.06 ppm of iron gave a moderate rust colored stain, while 0.04 ppm produced considerable brownish black stain. Generally speaking, the conditions of these tests were such as to accentuate the staining properties of the water, since considerably more water was evaporated over a unit area than would be the case in most instances in the field.

With respect to organic impurities in water, it is virtually impossible to determine from a chemical analysis if the water would cause objectionable staining when used for curing concrete. It is advisable to use a performance type test procedure, an example of which is Test

Method CRD-C 401-57 of the Corps of Engineers, U.S. Army [*23*]. This method of test outlines three procedures for evaluating the staining properties of water proposed for use in curing concrete. The Preliminary Method is intended for use in selecting sources that are worthy of more complete investigation and consists of evaporating 3000 ml of the test water in the concave area formed by the impression of a 4-in. watch glass in the surface of a neat white cement or plaster of Paris specimen. The Complete Method can be used to evaluate those sources that the Preliminary Method indicates to be promising. In the Complete Method, 3 gal of test water drips on to a mortar specimen exposed to heat lamps and forced circulation of air. The Field Method is intended as a means of evaluating the water finally selected for use and involves the curing of a 20-ft^2 slab of concrete with the test water under maximum exposure to the sun with the test slab placed at a slight angle to keep it in a wet condition with a minimum runoff. The test results by each of these three methods are evaluated by visual observation.

The latest Corps of Engineers Standard Practice Manual for Concrete [*24*] notes that the Standard Guide Specifications do not require that water be nonstaining. However, the manual does clearly state that there must be no permanent staining of surfaces where appearance is important. For these surfaces the contractor has the option of using nonstaining water or of cleaning the surface after completion of moist curing. No cleaning is required of surfaces which will subsequently be stained when the structure is in service.

SUMMARY

Mixing Water:

The significance of the foregoing information presented indicates that any naturally occurring or municipal supply waters suitable for drinking purposes can be used as mixing water for concrete and that most naturally occurring waters ordinarily used for industrial purposes are satisfactory. Many waters, which upon casual examination would be judged to be unsuitable because of color, odor, or contamination with impurities as in the case of marsh water, alkaline sulfate waters, and water containing industrial wastes, would be found to be satisfactory when tested in mortar or concrete since in many instances the strength would be greater than 85 to 90 per cent of the strength of comparative specimens made with pure waters. In the case of sea water, a strength reduction ranging from 8 to 15 per cent can be expected depending on job conditions; however, sea water is not ordinarily recommended for use as mixing water in reinforced concrete. The hardness of water does not affect air content of concrete, but algae can entrain air and significantly reduce strength.

Curing Water:

It is improbable that a water used for curing would attack concrete if it were of the type suitable for use as mixing water. Organic matter or iron in the curing water can cause staining or discoloration of concrete, but this is rather uncommon especially where a relatively small volume of water is used; however, the suggested performance tests [*21*] will determine if a water possesses any potential staining qualities.

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Other Materials

CURING MATERIALS

By C. E. Proudley,[1] Personal Member, ASTM

The environment of concrete immediately after being mixed and deposited in the forms, particularly with reference to moisture content and temperature, known as curing, is an important factor with respect to its ultimate strength and durability. Optimum curing conditions are essential to the attainment of the maximum potential quality of concrete. Minimum curing may produce adequate quality of concrete for the intended use. The difference between optimum and minimum curing acceptable for a job can be a debatable subject.

The influence of curing upon the performance of concrete has been the subject of study by a number of investigators and all have concluded that inadequate or improper curing will result in a variety of undesirable features, such as lower strength, random cracks, scaling, dusting, lack of resistance to abrasion, and lower resistance to weathering.

Curing is described briefly as keeping the concrete moist and warm enough that hydration of the cement can continue. More elaborately, it is "the process of maintaining a satisfactory moisture content and a favorable temperature in concrete during the period immediately following placement so that hydration of the cement may continue until the desired properties are developed to a sufficient degree to meet the requirements of service" [*1*].[2] This definition approaches that which can be considered minimum curing.

Curing is being given a place of increasing importance as the demand for high-quality portland cement concrete increases. From a position of total neglect or only a minor detail of concrete work it has risen to that of equal footing with the selection of proportions of the ingredients. This is entirely reasonable when it is recalled that standard test methods for making and curing test specimens for the design of a concrete mixture that will yield a prescribed strength require closely controlled temperature and moisture conditions throughout the laboratory curing period, ASTM Method of Making and Curing Compression and Flexure Test Specimens in the Laboratory (C 192 – 62 T).

There are a number of factors influencing the selection of the curing process to be used that depend largely upon the product under consideration. Useful information has been developed through research and observation over the years, both in the laboratory and under practical field conditions of job operation [*2*]. The effects of improper or inadequate curing are often difficult to identify, since there are other deleterious influences which may cause defects and disintegra-

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

tion similar in appearance to the effects of lack of curing.

TEMPERATURE EFFECTS

Hardening of concrete, or the attainment of strength, is usually of primary concern. The rate at which hardening occurs is influenced by the temperature of the concrete more than any other factor, not including composition of the portland cement or the concrete mix. Like most physicochemical reactions, an increase in temperature speeds up the hardening process [*3*]. Near the freezing temperature of water the rate of strength gain is so slow as to seriously interfere with construction progress. On the other hand, the temperature should not exceed about 160 F because of the danger of damaging the concrete (except for the autoclave curing of concrete products).

Heat resulting from the hydration of cement is ordinarily not a problem except in large massive structures where such measures as using a cement with low heat of hydration properties, cooling of materials, and circulating cold water in pipe coils embedded in the concrete become necessary. Heat of hydration is used to advantage during cold weather concreting [*4*].

Concrete at all times, whether in the hardened state or before, is subject to volume change stresses caused by temperature variations, hence the need to protect it from temperature fluctuations until it has developed sufficient strength to resist the stresses.

Ambient temperatures in any season of the year must be given careful attention. In those climates or locations where the 24 hr variation is more than 20 F, some protection against atmospheric temperature changes should be provided. Wood forms are adequate until the range goes beyond 35 F, under which circumstances a further insulating cover should be used to minimize random cracking and other defects that might be caused by rapid volume change or thermal shrinkage.

When concrete freezes ice crystals form, separating the solid constituents, so that when thawing occurs the mass is unconsolidated and spongy, resulting in hardened concrete that is porous and weak. It is very important to prevent freezing of concrete, and this may require not only insulation but the application of heat. For practical purposes it is well to consider that concrete freezes at the freezing point of water, that is, 32 F. At the present time there are no known antifreeze materials for addition to the concrete mixture that are not harmful. Calcium chloride (not over two per cent by weight of the cement) is used as an accelerator to hasten setting time and strength development, thereby reducing the period of susceptibility to freezing damage [*5*]. It is not an antifreeze.

Hot-weather concreting presents more difficult conditions to control, since the heat of hydration develops faster and insulation from atmospheric temperature is of little value unless it includes a means for removing heat. Under most circumstances the vapor pressure differential at high temperatures will cause moisture in the concrete to evaporate more rapidly than at the lower temperatures, in other words, drying out of the concrete above 80 F will remove the needed water quicker than at 50 F. Generally, the most effective means for protection on jobs where the temperature of the concrete may rise above 100 F due to the combined effect of temperature of the ingredients and the subsequent heat of hydration is to reduce the temperature of the ingredients initially so that the concrete will not exceed a maximum of 100 F during the curing period. Unless the concrete is handled in such manner that no excessive shrinkage stresses will develop during curing, there will be undesirable random cracks.

MOISTURE EFFECTS

The water available to the cement in the concrete has a major function in the development of the strength and other desirable characteristics [*6*]. For the purpose of this discussion it will be assumed that the specified amount of water was used and that bleeding (water gain) has been properly removed by delayed finishing or some acceptable process. It then remains to maintain throughout the mass of concrete a supply of water to provide for continuous hydration of the cement. In order that the desired properties be developed to the maximum as quickly as possible, it is essential that there be no interruption to the hydration process when once the reactions have started.

A deficiency of water will occur at surfaces exposed to the atmosphere. Inadequate moisture during curing will affect the quality of the surface first. The unhydrated cementing material at the surface will be apparent as dust and the weak bond will fail to hold the aggregate in place, resulting in a rough surface usually of irregular pattern.

It should be repeated that in order to secure maximum benefit from curing, the method used must assure adequate water to maintain continuous hydration of the cement throughout the mass at all times, until the desired strength and hardness have been attained.

CONTROL OF TEMPERATURE

The temperature of freshly mixed concrete for structures and pavements generally should be held between 50 and 80 F. This may require either heating the concrete during production or, in hot weather, cooling [*4,7*]. Because the specific heat of water is greater than that of any of the other ingredients, the temperature of the mixing water is given first consideration. Sometimes, however, the conditions require that temperature of the aggregates be raised or lowered as well.

Aggregates can be heated by various means such as stockpiling them over culvert pipe through which the heat from a fire is directed. Live steam is used, when available, by injecting it through small water pipes into several parts of the stockpile. If the materials are being handled through a batching plant the bins may be surrounded by steam coils and insulated. In any event, the procedure must be such as to secure a uniform temperature of the aggregates so that there will not be variations from batch to batch. Also, when steam injection is used the accumulation of condensed steam will affect the moisture content of the aggregate, which will require careful adjustment of batch weights to maintain uniform consistency and strength of concrete.

Cooling of water and aggregates during hot weather usually presents more problems than heating them during cold weather. If sufficiently low humidity prevails, the water temperature can be reduced in cooling towers by evaporation. Deep wells usually provide cooler water than city water supplies. Occasionally, crushed ice is included in the concrete mixture as part of the mixing water, and this is very effective. Aggregates may be cooled by keeping a fine spray of water on them by use of lawn sprinklers on the stockpiles.

METHODS OF CURING

Two general procedures for curing are in common use in the field: (*a*) an impervious sheet material or film to retain practically all of the mixing water in the concrete, and (*b*) a covering of wet material which compensates for or minimizes the loss of water through evaporation. Integral curing materials have not been developed, and, at the present time, additions of any substances into the fresh concrete as a substitute for protective coverings are not recommended. A third method is used extensively at concrete products manufacturing plants consisting of enclosures maintained at a relative humidity of nearly 100 per cent in which the molded units are placed. This method usually includes temperature control intended to accelerate the hardening process and accomplish the end result in a matter of hours instead of days. After the fresh concrete units have had an initial curing period of 2 hr, more or less, at the temperature of molding, wet steam is introduced into the chamber and the temperature surrounding the units gradually raised at a uniform rate of not more than 40 F/hr (60 F/hr for units of small size or thickness) until a maximum of 160 F is reached. After holding at the maximum temperature for a time determined by experiment to be required to develop the desired strength, the units are cooled slowly to avoid differential shrinkage cracking [*8*].

Impervious sheet materials and films are used principally where the supply of water is limited or where it requires an excessive investment in piping or water tank trucks to maintain wet covering as for paving. Liquid membrane-forming compounds, with or without a white or gray pigment, are sprayed on exposed surfaces of fresh concrete, and the film thus formed by rapid drying of the compound retards the loss of moisture from the concrete for several days or longer. The film subsequently flakes off. An asphalt emulsion or similar bituminous coating is good where the black color will not interfere with temperature control or appearance; the bituminous film will remain for a long time under most conditions.

Waterproof paper, reinforced with fibers and treated to reduce shrinkage, is effective in retarding moisture loss and may also provide a useful amount of temperature control. It is applied either directly over the concrete so that the concrete will not be marred, or it may be applied subsequent to a preliminary wet curing of fog spray or wet burlap for several hours.

Plastic film, such as polyethylene or combinations of the plastic with fibers or fabric for strength and durability, is used in the same manner as waterproof paper.

If there is an ample supply of water, such as at bridge construction over streams, the wet method of curing is used frequently. Burlap, cotton mats, sand, sawdust, earth, straw, and similar insoluble porous materials are spread over the fresh concrete when it has hardened enough not to be disturbed by the procedure and kept wet by sprinkling at frequent intervals so that free water is always available at the surface of the concrete. Ponding water on horizontal surfaces by use of dams of clay soil at the edge of the slab makes a satisfactory curing process. Nozzles to produce a fine spray or mist make excellent curing when temperatures are above 60 F and the wind is relatively calm.

EVALUATION OF CURING PROCEDURES AND MATERIALS

The effectiveness of various curing methods has been studied from time-to-time by means of experimental installations [*2*]. Interesting and valuable information has been obtained through many such studies. It would be thought that such organized investigations would be the logical means for determining the relative merits of different methods and materials, and, in fact, they can be. However, certain variables, such as relative humidity and ambient temperatures, are unpredictable and uncontrollable in the field under practical construction conditions, and the conclusions from these studies are always conditional. Many years ago Jackson and Kellermann stated: "It is believed that such comparisons should be made only in the laboratory under closely controlled temperature and humidity conditions, using a test procedure that will permit direct comparisons of the efficiency of different curing materials [*9*]." Limited comparisons of various methods under field conditions can be made, however, by following the procedure outlined in the Highway Research Board Circular 381 [*10*].

The importance or intended use of a structure may raise the question of the degree of curing efficiency that is most economical for the situation. It is obvious that, other circumstances being the same, less attention to curing will be necessary where the atmospheric conditions are moderate temperature with high-relative humidity, such as might prevail in a completely enclosed room of a building with temperatures ranging from 60 to 80 F. On the other hand, a slab exposed to the sun's rays with surrounding temperature of 100 F or higher and a relative humidity of 20 per cent or less should be given the maximum protection regardless of the service to which it will be exposed. Specifications can not be written to anticipate conditions during construction, in most cases; therefore, it is advisable to require curing protection considered to be adequate for the most severe conditions that might exist in the area covered by the specifications.

Among the conclusions to practically all studies of the effect of curing on the properties of concrete will be found statements that: (*a*) maintaining a moist condition in the concrete is essential to accomplish the desired results, such as, design strength, resistance to abrasion, and resistance to weathering, and (*b*) it is essential that the availability of water for the curing process be continuous from the time the concrete is mixed, since any interruption of the hydration of the cement will seriously retard further hydration and the attainment of the potential strength [*11,12*].

The efficacy of those curing methods and materials that make use of applied water as with burlap, cotton mats, sand, earth, and so on, is, obviously, dependent on the conscientious application of water throughout the curing period. The more absorbent materials, such as cotton mats and earth, are less likely to dry out to a serious extent between casual wettings than burlap, sand, or straw. There is no laboratory test of these materials acceptable for measuring their efficiency under service conditions other than measurements of weight per square yard of burlap and the examination of cotton mats for compliance with specifications for their fabrication. Competent visual inspection during use in the field is the key to success with these curing materials.

Impervious sheet materials, such as waterproof paper blankets and polyethylene film, may be tested in the laboratory using ASTM Test for Water Retention Efficiency of Liquid Membrane-Forming Compounds and Impermeable Sheet Materials for Curing Concrete (C 156 – 64 T) for measuring the efficiency of water retention or impermeability; however, these materials, when new, will nearly always meet the minimum requirements. After use it may be found that leakage of water vapor through these sheet materials takes place due to damage in handling and from walking or operating equipment on the hardened concrete while it is still being cured. Tears, cracks, pin holes, and punctures will permit an important amount of curing water to escape unless the imperfections are repaired properly and promptly. Sheet materials must be spread to cover all parts of the structure not covered by forms, and a tight seal must be secured at all edges and joints of the sheets. On horizontal surfaces this can be accomplished with windrows of earth at the edges where the sheets are lapped.

Liquid membrane-forming compounds must be tested in the laboratory before use in the field and, in addition, their application must be carefully supervised on the job. Again, ASTM Method C 156 is used to determine the water retention efficiency when applied in the required amount to laboratory test specimens. Application to the structure must be at the specified rate of coverage in gallons per square yard, and this must be uniform. Specifications for curing compounds (ASTM Specification for Liquid Membrane-Forming Compounds for Curing Concrete (C 309)) include requirements for other properties such as drying time, light reflectance for pigmented compounds, sprayability, and storage stability. Pigmented compounds are used extensively for curing pavement to reduce the absorption of solar heat, thus minimizing the surface temperature as well as the internal temperatures. Field inspection to assure that the pigment is completely in suspension when applied is especially necessary when the labor is inexperienced.

The wet curing methods generally provide a good degree of temperature control for hot-weather work. Imprevious sheet materials reflect solar heat to a moderate extent, provided the material is light in color, such as white polyethylene or natural color kraft paper. Any dark colors, including bituminous applications, are objectionable except when used in areas protected from the sun's rays.

DURATION OF CURING

Concrete kept continuously wet will continue to increase in strength and hardness over a period of years. For practical reasons the length of time that a curing procedure may be reasonably required is confined to that which will assure that the properties required of the concrete have been attained or will be reached before service conditions are imposed on it. Where sheet materials, sprays, or temporary coverings are used they are kept on for 3 to 14 days; generally a minimum of 3 days for pavements and 14 days for structures, unless a high early strength cement has been used, in which case the curing periods may be shortened to half or even one third of this time. Membrane-forming compounds remain on the concrete as a dry film until they are brushed off intentionally for subsequent operations, or until natural forces remove them. The formulation of compounds should be such as will assure their flaking off within several months or less, especially where surfaces are exposed to public view. The effectiveness of the sprayed-on film for curing gradually diminishes to practically zero in a matter of several weeks to months depending on the nature of the solids and solvents used in the curing compound and the conditions of exposure.

The curing period cannot be prescribed definitely when temperatures range below 40 F. Cement reactions are so slow at the lower temperatures that extra time must be allowed. This is usually specified by stating that any day on which the average temperature surrounding the concrete falls below 40 F shall not be counted as a day of curing. This requirement suggests temperature protection and the application of heat by various artificial means, such as heated enclosures. In such enclosures care must be exercised that the temperature is practically uniform and without hot spots at any part of the curing area. It is cautioned, also, that when a source of heat is used that produces carbon dioxide, that the fumes be vented to the outside since carbon dioxide (CO_2) will combine with the moist cement to form calcium carbonate and, thereby, result in a soft and dusty surface even though otherwise properly placed, trowelled, and cured.

TESTS AND THEIR SIGNIFICANCE

Based on the premise that the efficiency of a curing material can be determined by its ability to retain moisture in the concrete, Method C 156 has been published by the American Society for Testing and Materials. The method measures the loss of moisture from mortar specimens during the first 72 hr when held at 100 ± 2 F and relative humidity of 32 ± 2 per cent. The maximum loss allowed by ASTM Specification for Waterproof Paper for Curing Concrete (C 171) and by ASTM Specification for Liquid Membrane-Forming Compounds for Curing Concrete (C 309) is 0.055 g/cm^2 (slightly more than one pint per square yard). This loss during the first three days will not affect the quality of the concrete adversely.

Specifications for waterproof paper for curing include requirements for wet tensile strength to be determined by ASTM Test for Wet Tensile Breaking Strength of Paper and Paperboard (D 829). This is for the purpose of securing reasonable serviceability for possible reuse of the paper blankets.

Liquid membrane-forming compounds for curing, if pigmented, are required to have a daylight reflectance when tested by ASTM Test for 45-Deg, 0-Deg Directional Reflectance of Opaque Specimens by Filter Photometry (E 97), of not less than 60 per cent of that of magnesium oxide if white and not less than 50 per cent if gray pigmented. This provides for a large degree of temperature control in the concrete through the reflection of solar heat. No reflectance requirement is given for clear or translucent compounds or for black curing materials, it being assumed that other means will be used to control the concrete temperature.

The membrane-forming compounds must have a consistency that will permit them to be sprayed readily at 40 F, and the consistency must also assure adherence to vertical concrete surfaces at the specified rate of application without running or sagging. Drying time is limited to not more than 4 hr under standard laboratory conditions of 73.4 ± 3 F and 50 ± 10 per cent relative humidity in an air current of 600 ft/min or more. This requirement is to take care of early damage by rain, dust, and debris. It must not be tacky when dried, as this would make it likely to pick up if touched or walked on, thus breaking the seal.

A precautionary paragraph is included in the specifications against formulations containing benzene, toluene, carbon tetrachloride, methyl alcohol and other toxic or dangerously flammable constituents. The formulation must also be free from ingredients which react unfavorably with the cement to cause a weak mortar surface. To test for this, a quick approximation is recommended through a scratch test with a metal point to compare the hardness of the membrane cured surface with a similar concrete surface that has been cured with water only.

Most construction specifications and contracts are adequate in the matter of providing appropriate curing for the concrete work, if they are followed intelligently and faithfully. Curing operations are the final step in the concrete construction, and the inspection of this phase of the work is often reduced to the minimum or left to the workmen who are last to leave the scene. The needless expense that may be incurred for repairs resulting from inattention to curing details should be apparent to every supervisor and inspector.

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[*6*] T. C. Powers, "A Discussion of Cement Hydration in Relation to the Curing of Concrete," *Proceedings*, Highway Research Board, Vol. 27, 1947, pp. 178–188.

[*7*] "Recommended Practice for Hot Weather Concreting," *Standard 605-59*, Am. Concrete Inst., 1963, pp. 605-1 to 605-10.

[*8*] "Low Pressure Steam Curing," *Journal*, Am. Concrete Inst., August, 1963.

[*9*] F. H. Jackson and W. F. Kellermann, "Tests of Concrete Curing Materials," *Public Roads*, Vol. 20, No. 4, June, 1963.

[*10*] "Recommended Procedures for Determination of Relative Merit of Curing Methods for Portland Cement Concrete Pavements," *Circular 381*, Highway Research Board, March, 1959.

[*11*] T. Waters, "Effect of Allowing Concrete to Dry Before It Has Fully Cured," *Magazine of Concrete Research*, England, Vol. 7, No. 20, July, 1955, pp. 79–82.

[*12*] H. J. Gilkey, "Moist Curing of Concrete," *Engineering News-Record*, Vol. 119, No. 16, Oct. 14, 1937, pp. 630–633.

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Other Materials

AIR-ENTRAINING ADMIXTURES

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The use of intentional air entrainment in concrete is now a well-established means for greatly enhancing the ability of concrete to resist the potentially destructive effect of freezing and thawing. Its use should be mandatory when concrete is to be exposed to such an environment, particulary when chemical deicers are being used, as on pavements and bridge decks.

A thorough survey of the early development of air entrainment is presented by Gonnerman [*1*].[2] The following paragraph from Gonnerman's report is of particular significance:

> These projects (test roads constructed in 1935–1937) revealed no relationship between surface scaling and composition of the cement, but they did show clearly that portland cement that inadvertently contained "crusher oil" reduced surface scaling as did many of the blends of portland and natural cement that contained tallow added during grinding of the natural cement. Laboratory tests disclosed that the beneficial effect of the crusher oil and tallow was due entirely to the additional air entrapped in the concrete by these air-entraining agents.

Other investigators [*2–6*] came to similar conclusions during the late thirties and early forties.

In these early instances, the air entrainment was not intentional but resulted from the presence of the crusher oil or the use of the tallow as a grinding aid during the production of the cement. These were the forerunners of the materials now used to produce air-entraining cements and called additions. Materials similar to presently used additions are called air-entraining admixtures when added with the other concrete ingredients at the time of mixing. This report is concerned with this class of materials. What are these materials; how do they function, both as to the process of entraining air and enhancing durability; how can they be specified and tested to insure adequate performance? These are some of the questions which we will attempt to answer in this chapter.

Definitions:

It will be helpful to define certain of the terms used in discussing air entrainment. This listing is by no means complete, but the following definitions appear appropriate:

Air Entrainment—The introduction of air in the form of discrete air voids or bubbles dispersed throughout the mixture as a result of the use of air-entraining materials.

Entrained Air—The air, made up of discrete air voids, which becomes part of a mixture during the process of air entrainment.

Entrapped Air Voids—Air voids not re-

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

sulting from intentional air entrainment. Such voids are larger than those resulting from intentional air entrainment and are at times referred to as natural air voids.

Entrained Air Voids—Air voids resulting from the use of intentional air entrainment. Such voids are generally spherical in shape and considerably smaller than the natural air voids.

Air-Entraining Admixture—A material added to cementitious mixtures at the time materials are batched for mixing, the use of which results in intentional air entrainment. (See ASTM Definition of Terms Relating to Concrete and Concrete Aggregates (C 125) for the definition of an admixture.)

Air-Entraining Addition—Air-entraining material interground with, or to be interground with, hydraulic cement. (See ASTM Definition of Terms Relating to Hydraulic Cement (C 219) for the definition of an addition.)

Air-Entraining Agent—A material the use of which results in intentional air entrainment when included in a mixture; a term that should be used only when it is intended to refer to materials that can be used both as air-entraining additions or admixtures.

MATERIALS USED AS AIR-ENTRAINING ADMIXTURES

There are many materials capable of functioning as air-entraining admixtures. In an extensive evaluation program, the Bureau of Public Roads [*7*] separated 27 commercial air-entraining admixtures submitted for test into the following classifications: (1) salts of wood resins (pine wood stumps), (2) synthetic detergents (petroleum fractions), (3) salts of sulfonated lignin (paper pulp industry), (4) salts of petroleum acids (petroleum refining), (5) salts of proteinaceous materials (processing of animal hides), (6) fatty and resinous acids and their salts (paper pulp and animal hide processing), and (7) organic salts of sulfonated hydrocarbons (petroleum refining). Of these 27 materials, 17 were liquids, 5 were powders, 4 were in flake form, and 1 was semisolid.

Some materials, such as hydrogen peroxide and powdered aluminum metal, can be used to entrain gas bubbles in cementitious mixtures but are not considered as acceptable air-entraining admixtures, since they do not necessarily produce an air void system which will enhance resistance to freezing and thawing.

Generally, only small quantities of air-entraining admixtures are required to entrain the desired amount of air. These are of the order of 0.05 per cent of active ingredient by weight of the cement.

FUNCTION OF ENTRAINED AIR

The major reason for the use of intentionally entrained air is to provide concrete with a high degree of resistance to freezing and thawing and the use of chemical deicers. (The discussion to follow will also be applicable to the use of air-entraining cements, in which the air-entraining agent or material is used as an addition during the grinding of the cement clinker.) There are numerous other advantages, also, to the use of intentionally entrained air. For example, plasticity and workability are increased, enabling a reduction in water content. Uniformity of placement and consolidation can be achieved more readily, thus reducing segregation, and bleeding is reduced. These and other advantages are discussed in detail in a number of the appended references, for example [*8,9*].

To achieve the improvement in frost resistance, the intentionally entrained air must have certain characteristics. Not only is the total volume of importance, but more importantly the size and distribution of the air voids must be such as to provide efficient protection to the cement paste. Powers' [*10–12*] contributions to the understanding of how entrained air functions in providing increased frost resistance have been outstanding.

TABLE 1—CHARACTERISTICS OF CONCRETES AT OPTIMUM[a] AIR CONTENT.
(Tables 17 and 18, Ref. *15*)

Max Size of Aggregate	Cement Content: 4 bags per cubic yard					Cement Content: 5½ bags per cubic yard					Cement Content: 7 bags per cubic yard				
	Total Air Content, per cent	Mortar Air Content, per cent[b],	Paste Air Content, per cent[b]	Bubble-Spacing Factor, in.[c]	Net Water-Cement Ratio, gal per bag	Total Air Content, per cent	Mortar Air Content, per cent[b]	Paste Air Content, per cent[b]	Bubble-Spacing Factor in.[c]	Net Water-Cement Ration, gal per bag	Total Air Content, per cent	Mortar Air Content, per cent[b]	Paste Air Content, per cent[b]	Bubble-Spacing Factor, in.[c]	Net Water-Cement Ratio, gal per bag
2½ in.	4.5	8.8	18.5	0.012	6.30	4.5	9.1	16.7	0.007	4.75	4.5	9.2	14.7	0.007	4.05
1½ in.	4.5	8.3	18.2	0.013	6.60	4.5	8.5	16.4	0.008	4.75	4.5	8.4	14.3	0.008	4.05
¾ in.	5.5	8.9	19.8	0.013	7.55	5.0	8.3	16.9	0.009	5.25	5.5	9.2	16.8	0.007	4.30
⅜ in.	8.5	11.1	26.2	0.009	8.50	6.5	8.7	19.7	0.011	6.00	7.0	9.6	19.4	0.008	4.75
No. 4	12.5	12.5	31.6	0.006	10.30	9.0	9.0	23.0	0.012	7.55	10.0	10.0	23.4	0.008	5.80

[a] Optimum determined from relation between expansion during 300 cycles of freezing and thawing and air content of concrete.

[b] Calculated as follows:

$$\text{Mortar air content, per cent} = \frac{A}{C + W + S + A} \times 100$$

$$\text{Paste air content, per cent} = \frac{A}{C + W + A} \times 100$$

where:
C = absolute volume of cement,
W = volume of net mixing water,
S = absolute volume of fine aggregate (saturated surface dry), and
A = volume of air.

[c] Calculated from linear traverse data using method outlined in *Bulletin No. 33*, Portland Cement Assn. [*11*].

This work developed the concept of internal hydraulic pressure created by the resistance to flow or movement of excess water volume produced during the freezing process as being the mechanism responsible for distress. To keep this internal pressure below the tensile or rupture strength of the paste, Powers showed that the air voids must be well distributed throughout the matrix (cement-water paste component) and sufficient in number so that each void provides protection to the cement paste surrounding it, and the protected volumes overlap to leave no unprotected paste. Each void provides space into which the excess water can move during freezing, and the voids should be sufficiently close to one another so as to avoid high internal hydraulic pressure in the paste during movement of water to the air void.

Later work by Powers and Helmuth [*13*] indicated that, in addition to the generation of hydraulic pressure during freezing, another important factor may be the diffusion of gel water to capillary cavities contributing to the growth of ice bodies in these cavities resulting in the development of expansive forces. This factor may be of significance in nonair-entrained concrete. The presence of an adequate air-void system provides spaces containing ice which competes with the ice in the capillary cavities for this unfrozen gel water, and thus avoids the development of these expansive forces.

Powers [*11*] developed the concept of void spacing factor to characterize an air-void system and analyzed laboratory freezing and thawing data available at that time to show that the void spacing factor for frost resistance should be about 0.01 in. or less. The void spacing factor is defined by Powers as the average maximum distance from a point in the cement paste to the nearest air void. This is an indication of the distance water would have to travel, during the freezing process, to reach a protective air void. More recent work by Mielenz et al [*14*] indicates that an upper limit of about 0.006 to 0.008 in. is required for extreme exposures. Extensive freezing and thawing tests by Klieger [*15*] provided further substantiation of the void spacing factor concept. These and other tests [*16*] called attention to the need for different volumetric air-content requirements for concretes made with different maximum sizes of coarse aggregate. Table 1 shows this effect of maximum size of aggregate on the optimum air content, along with void spacing factors. Although the total air contents of the mixtures shown in Table 1 vary through a wide range, the air content of the mortar fraction is essentially constant at about 9 per cent. As the maximum size of coarse aggregate increases and consistency and cement content held constant, less mortar is required in the mixture [*15*]; therefore, change in total concrete air content with change in maximum size of coarse aggregate is to be expected.

Most of the early field and laboratory work on air-entrained concretes dealt with paving-type concrete in which the coarse aggregate was generally about $1\frac{1}{2}$-in.-max size. In such concretes without intentional air entrainment, the air content (composed of entrapped air voids which are too large to be effective with respect to improving frost resistance or workability but which are necessarily included when air content is expressed in volumetric terms) may range up to as high as 1.0 or 2.0 per cent. Field and laboratory tests indicated that when such concretes were provided with about 3 per cent additional entrained air the resistance to freezing and thawing and deicer chemicals was greatly enhanced. This led to the specification that such concrete should contain 4.5 ± 1.5 per cent air, based on the premise that the coarse aggregate would have a maximum size of $1\frac{1}{2}$ in. and that the average amount of entrapped air would be about 1.5 per cent and composed of relatively large and ineffective air voids. For these concretes, the provision of an additional 3.0 per cent of intentionally entrained air by the use of an air-entraining admixture provided an air-void system well distributed throughout the matrix and containing a sufficient number of air voids to meet the void spacing factor requirements which later studies demonstrated as necessary.

Specifications based on volume of entrained air still remain the only practical way of specifying intentionally entrained air, despite the fact that other parameters are of more importance than volume alone. Examples of such use are found in American Concrete Inst. (ACI) Standard "Recommended Practice for Selecting Proportions for Concrete (ACI 613-54)" and specifications of agencies such as the Corps of Engineers, U.S.

Army, and the Bureau of Reclamation. For example, the Corps of Engineers, U.S. Army, "Guide Specifications for Concrete," CE 1401.01, October, 1953, Section -08c, "Entrained Air Content," states:

> The total calculated air content of that portion of the concrete containing aggregate smaller than the 1½-in.-square mesh sieve shall be between 4 and 7 per cent of the volume of the concrete based on measurements made on concrete immediately after discharge from the mixer.

TABLE 2—INFLUENCE OF AIR-VOID CHARACTERISTICS ON RESISTANCE OF CONCRETE TO FREEZING AND THAWING.

Air-Entraining Admixture	Air Content, (C 231), per cent	Air-Void Characteristics (ASTM Recommended Practice C 457)					Freezing and Thawing Cycles for 0.10% Expansion
		Air, per cent	Number of Voids/in.	Void Spacing Factor, in.	Specific Surface, in.^{2}/in.^{3} of air	No. of Voids/in.^{3} Concrete, millions	
None	1.8	1.1	0.8	0.031	302	0.08	19
A	6.0	4.1	4.0	0.013	387	0.11	29
B	6.0	4.1	4.9	0.010	480	0.22	39
D	5.0	3.2	3.3	0.013	416	0.26	82
E	5.8	3.5	5.1	0.009	577	0.78	100
F[a]	5.2	3.9	9.6	0.006	990	3.79	550

[a] A commercial air-entraining admixture meeting the requirements of ASTM Specification C 260.

Although this was directed primarily to mass concrete, it was a recognition of the need for considering the effect of the maximum size of coarse aggregate on the required air content of concrete.

Specifications and control tests will continue to be based on the volume of air entrained in the concrete, rather than on the size and distribution of the air voids in the cement-paste matrix, until a means is developed for readily determining other air void parameters directly on the freshly-mixed concrete in the field. It would be highly desirable to have a test method available that could provide a measure of size and distribution of air voids within a few minutes after completion of mixing. Not having such a test method available, it is indeed fortunate that concretes having total air contents in the range of the optimum air contents shown in Table 1 will have the proper size and distribution of air voids when the air-entraining admixture used meets the requirements of ASTM Specification for Air-Entraining Admixtures for Concrete (C 260 – 65 T).

The importance of size and distribution of air voids, as contrasted with total volume of voids alone, can be seen in the results of a study made in the laboratories of the Portland Cement Assn. Air-entrained concretes were prepared using an acceptable proprietary air-entraining admixture and four nonproprietary materials which exhibited a potential for entraining air. A nonair-entrained concrete was also included in these tests. In addition to the determination of air content of the freshly-mixed concrete as described in ASTM Test for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231), the air content, void spacing factor, specific surface, and number of voids per lineal inch of traverse were determined on the hardened concretes as described in ASTM Recommended Practice for Microscopical Determination of Air-Void Content, Specific Surface, and Spacing Factor of the Air-Void System in Hardened Concrete (C 457 – 60 T). Additional refinements of the technique enabled the determination of the total number of air voids per unit volume. The results of these measurements and the performance of the concretes when frozen and thawed while immersed in water are shown in Table 2.

It is apparent from these data that an air-entraining admixture must not only be capable of entraining some volume of air but also that the air void system must be characterized by a large number of small, well distributed air voids in order to provide a high degree of frost resistance.

FACTORS INFLUENCING AMOUNT AND CHARACTER OF ENTRAINED AIR

The works of Mielenz et al [*14*] and Bruere [*9,17–19*] have made significant contributions to the understanding of the mechanism by which air-entraining admixtures function and the influence of a number of different variables. Mielenz and his co-workers dealt extensively with the origin, evolution, and effects of the air-void system in concrete. They showed that in the concentrations normally used in concrete, air-entraining admixtures are positively adsorbed at air-water interfaces and that the surface tension of the water is decreased about 25 per cent. This adsorption at air-water interfaces produces a "film" of air-entraining admixture which influences the air-retention properties of discrete bubbles formed during mixing. For some air-entraining admixtures, the calcium salt of the active constituent in the admixture may be only slightly soluble in water. In such instances, the film at the air-water interface may include a precipitated solid or gelatinous film enclosing each air bubble. Admixtures which produce a relatively insoluble precipitate in portland cement concrete include sodium soaps of wood resin, such as neutralized Vinsol resin and sodium abietate; sodium soaps of lignin derivatives, rosin, or fatty acid; or triethanolamine salts of sulfonic acid. Many calcium salts of sulfonic acids are soluble in water and many air-entraining admixtures in which the surface-active constituent is a sulfonate would not form such precipitated films around the air bubbles.

The amount and character of the air entrained in concrete is influenced by numerous factors, some of which are: (1) concentration of the air-entraining admixture and its influence on surface tension; (2) time of mixing; (3) speed of mixing, that is, rate of shearing action; (4) consistency of mixture; (5) temperature of the mixture; (6) water-cement ratio and water content of the mixture; and (7) the gradation of the solids in the mixture, including the cement. Mielenz et al [*14*] have concluded from theoretical considerations of pressure in air voids due to hydrostatic pressure of overlying concrete and the curvature of the air-water interface (that is, bubble size) and the solubility of air bubbles in water that both the total volume of air and the size distribution of the air bubbles can change in the unhardened concrete due to interchange of air between bubbles and dissolution of air. Their tests on air-water foams and dilute cement pastes tended to corroborate these conclusions. Bruere [*19*], however, has shown that such changes do not take place to any significant degree in air-entrained pastes after cessation of mixing, although such interchange is of significance during the mixing process and may be for a few minutes thereafter.

Further information on the influence of numerous variables on the characteristics of air entrained in concrete is of interest and is presented in the following paragraphs.

Type and Amount of Air-Entraining Admixture:

Mielenz and his co-workers theorize that the type of organic material which constitutes the active ingredient in the air-entraining admixture influences the amount and character of entrained air voids by its effect on: (1) surface tension; (2) the elasticity of the film at the air-water interface, (3) transmission of air across the air-water interface, and (4) adhesion of the air bubbles to particles of cement or aggregate. All of these factors will be operative during the mixing operation.

At the same volumetric air content, different air-entraining admixtures will produce air-void systems having different specific surfaces, number of air voids per unit volume, and spacing factors. Part of the differences occur during the mixing operation and part during handling, placing, and compaction. The potential importance of these differences with respect to freezing and thawing durability was illustrated by the data in Table 2.

Increasing the amount of acceptable air-entraining admixtures will increase the volume of air entrained, increase the specific surface of the air-void system, and decrease the spacing factor.

Fine Aggregate—Changes in grading of sand may alter the volume and nature of air in the mortar [*20*]. Appreciable increase in the quantity of very fine particles of sand will decrease the amount of entrained air and may reduce the maximum and median size of the individual air bubbles [*21*]. Appreciable increase in the quantity of the middle sizes of sand will tend to increase the air in the mortar. Sand gradation is of more importance in leaner mixes. In the richer mixes, the influence of gradation on entrained air is not as marked.

Sands have been reported as containing organic matter difficult to remove by ordinary measures which cause entrainment of large quantities of air in concrete. Such sands are relatively rare, but concretes made with them illustrate the importance of the distribution of the air as compared with simple volume. Macnaughton suggests [*22*] this type of entrained air be termed "accidental air" and since "it may be unstable in nature and vary in size . . . [it] should not be confused with . . . 'entrained air'."

Cement—As the cement content increases, the air entraining potential of an admixture will tend to diminish, and an increase in the fineness of cement will result in a decrease of the air entrained in the mortar [*23–25*]. Some regular (nonair-entraining) cements naturally entrain more air than others, and these require less air-entraining admixture to develop a given mortar air content.

Water—Increase in water-cement ratio is likely to result in an increase in air content. Although the volume of air entrained may increase, the specific surface of the air voids generally decreases and the spacing factor increases [*14*]. Nondegradable detergents present in water can result in excessively high and variable air contents.

Consistency—Within the normally used range, increase in initial slump is accompanied by an increase in air content in concrete mixtures [*8,23,25*]. Recent work by Klieger [*16*] indicates that the optimum mortar air content remains at about 9.0 per cent.

Mixing—The effect of mixing action on the amount of air entrained varies with the type and the condition of the mixer [*8,26*]. The amount of air entrained by any given mixer will decrease appreciably as the blades become worn, or as the mixing action is impaired if hardened mortar is allowed to accumulate in the drum and on the blades. An increase in entrained air will occur if the mixer is loaded to less than rated capacity, and a decrease will result from overloading the mixer. A stationary mixer, a paving mixer, and a transit mixer may develop significant differences in the volume of air entrained in a given concrete mixture. The air content will increase with increased time of mixing up to about 2 min in stationary [*8*] or paving mixers (and to about 15 min in most transit mixers), after which the air content may remain approximately constant for a considerable period before it begins to drop off. The reduction in air may result from an increase in very fine particles in the mixture with prolonged mixing action or simply from an increase in the ratio of air-escape to foam-generation in the latter portion of the mixing period. Recent Portland Cement Assn. tests of transit-mixed concrete indicate that the air void system, as characterized by specific surface and spacing factor, is not harmed by prolonged agitation.

Temperature—For a constant amount of air-entraining admixture, less air will be entrained at 100 F than at 70 F and more will be entrained at 40 F. In other words, everything else being equal, air entrainment varies inversely with temperature [*8,24*].

Vibration—Intensive internal vibration applied to concrete will cause air bubbles to rise to the surface and be expelled. The larger natural voids are most readily expelled [*14,27–29*]. Moderately small bubbles may tend to work upward if the vibration is intense and prolonged. There is increasing evidence, however, that the critically important spacing of small entrained-air bubbles in the matrix is disturbed very little, even by intense vibration. If vibration is applied as it should be, with just enough intensity and duration to effect consolidation, and if the mixture is designed properly, removal of the effective portion of the entrained air will not occur. In some instances externally applied vigorous vibration may cause an increase in air content. However, in this case the added air is in the form of relatively large natural voids.

Admixtures—Solid, finely divided admixtures such as fly ash or bentonite will reduce the air content. The effect becomes marked with fly ash as the carbon content increases. Calcium chloride can be used successfully with air-entraining admixtures, but if added in direct contact with certain common admixtures, a rapid chemical reaction may occur between the two, largely inhibiting air entrainment. On the other hand, some tests have indicated that when the inhibiting reaction does not take place, the air content may be increased about one per cent. Therefore, especial care must be taken when other admixtures are used in conjunction with air-entraining admixtures.

One of the most frequent and pronounced causes for variations in air content results from variation in the amount, type, or condition of the air-entraining admixture itself. This last cause, as with many of the other variations in concrete, is a function in turn of the alertness and adequacy of the control and inspection given the work.

Any influence which would maintain or even actually improve the distribution of the bubbles within the matrix or increase the ratio of air-boundary surface to air volume would be desirable. It is certain that an improved surface-to-volume ratio would reduce considerably the total volume of air needed to be entrained compared with what is now considered normal for optimum results.

Work under way in the research laboratories of the Portland Cement Assn. [*30*] and elsewhere points toward advances in the comprehension of the mechanics of air entrainment which should further establish this major advance in the technology of concrete. For the time being, however, evaluation must continue for the most part along the indirect lines of volume of air in the total mixture.

METHODS FOR DETERMINING AIR-VOID CHARACTERISTICS

Freshly-Mixed Concrete:

Since parameters such as specific surface and spacing factor of entrained air are more reliable criteria of effectiveness than the volume of entrained air, the ideal test would be one which could measure these characteristics in the freshly-mixed concrete. Unfortunately, no such method has yet been developed. For field control purposes, presently available test techniques can determine with reasonable accuracy only the volume of entrained air.

The gravimetric method described in ASTM Test for Weight Per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (C 138), is a simple test to make. It is based on the relation of actual to calculated air-free unit weight of the concrete and produces reasonably accurate results when aggregates of relatively uniform specific gravity are used. However, errors attributable to inadeate sampling may be introduced where the specific gravities of the fine and coarse aggregate differ materially or where either aggregate is itself composed of particles of materially differing densities.

The most widely used method is that described in ASTM Method C 231 which is a procedure for determining the air content of freshly mixed concrete by application of pressure. Based on Boyle's law, that at a constant temperature the volume of a given quantity of any gas varies inversely with the pressure to which the gas is subjected, the method is generally adequate for use with all ordinary types of mortar or concrete containing reasonably dense aggregate. Large errors will be introduced where highly vesicular or porous aggregates are used due to the impracticability of differentiating between the air in the aggregate particles and the entrained air in the paste. There are a number of pressure meters on the market which, by this method, produce satisfactory results with concrete mixtures containing aggregate up to about 2 in. in maximum size. When larger aggregate is used, the larger particles may be removed by hand and the effect of such a removal calculated in arriving at the total air content [*31*]. Passing samples of freshly-mixed concrete over a sieve to exclude large particles may lead to important errors resulting from loss of air during the screening operation.

The volumetric method described in ASTM Test for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173) is most useful for determining the air contents of concretes made with lightweight aggregates [*32*]. This technique eliminates the possibility of significant errors in differentiating between air in the aggregate particles and air in the paste.

Hardened Concrete:

The important characteristics of the entrained-air voids can most readily be determined in hardened concrete by microscopic examination of sawed and ground surfaces of the hardened concrete. ASTM Method C 457 is a procedure for determining the total air volume, specific surface, and spacing factor of air voids by either a linear traverse method or a modified point-count method. Additional refinements of the linear traverse equipment enable the measurement of chord-size distribution of air voids from which the total number of air voids per unit volume can be calculated, as shown in Table 2.

Mielenz and his co-workers [*14*] show the results of measurements of air-void characteristics of cores taken from a wide variety of structures. Although such measurements are time consuming, they can provide reassuring evidence of the effectiveness of the air-entraining admixture in providing the desired air-void system.

The volume of air in hardened concrete can also be determined by a high pressure method developed by the Illinois Highway Dept. [*33*].

CURRENT SPECIFICATIONS FOR AIR-ENTRAINING ADMIXTURES

It was realized rather early in the development of air entrainment that the size and distribution of the air voids could be expected to be of major importance with respect to the effectiveness of the entrained air in enhancing durability. Since there was no ready and reliable means for determining these air-void characteristics and since there was a need to evaluate the influence of these air-entraining materials on other concrete properties, a performance type specification was developed in 1950 by ASTM.

ASTM Specification C 260 evaluates the effects which any given air-entraining admixture under test may exert on the bleeding, compressive and flexural strength, resistance to freezing and thawing, strength of bond to steel, and the length change on drying of a concrete mixture in comparison with a similar concrete mixture containing a reference air-entraining admixture. The methods by which these effects may be tested are given in ASTM Method of Testing Air-Entraining Admixtures for Concrete (C 233 – 65 T). The criteria of Specification C 260 are assumed to afford assurance that if, under the particular conditions of the specified mixtures and conditions in Method C 233, the particular sample of the admixture under test exerts satisfactory influence on certain properties of the laboratory concrete, it will be reasonable to expect that the quantity of the air-entraining admixture represented by the sample will develop satisfactory entrainment of air in field concrete without deleterious effects. However, the scope of Method C 233 acknowledges that the

> . . . tests are based on arbitrary stipulations permitting highly standardized testing in the laboratory and are not intended to simulate actual job conditions.

All of the elaborate testing required by ASTM Specification C 260 reduces simply to a very indirect method of determining whether: (1) the particular admixture under test will produce relatively stable air voids which will become widely dispersed throughout the matrix of field mortar or concrete so as to produce an air-void system having the proper characteristics for enhancing durability, and (2) that the admixture contains nothing which will have a deleterious chemical effect on such mixtures. This specification, with its attendant test methods, while rather elaborate, has provided a means for evaluating air-entraining admixtures on a performance basis. The wide variety chemically of materials which can function as satisfactory air-entraining admixtures precludes the inclusion of chemical requirements.

In recognition of the fact that the tests required are elaborate and time-consuming and that consequently they may not be performed as often as desirable to ensure conformance, attention is being given to (1) modifications which will include requirements for insuring that subsequent lots of admixture are identical to the sample tested under the provisions of ASTM Specification C 260 and (2) adding requirements for uniformity of the admixture, including sampling and inspection procedures.

A further consideration which is receiving attention is the possibility that the freezing and thawing tests, which are probably the most costly and time-consuming part of the testing procedure, can be supplanted by an examination of the characteristics of the air-void system produced by the admixture under test and a comparison with the system produced by the reference admixture. ASTM Method C 457 provides the means for such a comparison; however, further information is needed on the accuracy and reproducibility of the method. Such information is being developed by ASTM Committee C-9, which is responsible for the various ASTM specifications and test methods to which this report has referred. Until that time, however, the job performance of an air-entraining admixture must still be based on the direct measurement of "total air content" and on its effect on other readily measured properties, such as slump, bleeding, and strength. In this respect, it is fortunate that the simple air-entraining admixtures available so economically in the United States appear to produce, in almost all cases, about the optimum air-void system in the matrix when the total air contents shown in Table 1 are present in the concretes.

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Other Materials

MINERAL ADMIXTURES

By E. C. Higginson,[1] Personal Member, ASTM

Admixtures are generally used to provide an economical means of improving one or more properties of fresh or hardened concrete. Among the effects sought are: reduction in bleeding, increased workability, acceleration or retardation of hydration or setting, increased strength, reduction in heat of hydration, added resistance to freezing and thawing, increased impermeability, improved resistance to aggressive waters and soils, and reduction of expansion caused by reactive aggregate and alkalies in cement. It should be noted that many of these improvements may be obtained without the use of admixtures if proper steps are taken in the design of concrete for the desired uses. However, admixtures designed for specific uses may effect the desired improvements in properties at a lower cost. Economy is, therefore, one of the matters of prime consideration.

Admixtures are defined under ASTM Definitions of Terms Relating to Concrete and Concrete Aggregates (C 125) as materials other than water, aggregates, and portland cement (including air-entraining portland cement and portland blast-furnace slag cement) that are used as ingredients for concrete and are added to the batch immediately before or during mixing. A wide variety of materials are so classified. Among these are organic compounds, such as triethanolamine, sulfonated lignins, oils, fats, resins, and carbohydrates; inorganic compounds, such as calcium chloride, borax, sodium carbonate, and sodium silicate; and finely divided minerals. Only mineral admixtures are discussed in this paper.

Mineral admixtures include any essentially insoluble material other than cement and aggregate, which is used as an ingredient for concrete, and is added to the batch immediately before or during mixing. Mineral admixtures include natural materials, processed natural materials, and artificial materials. They are finely divided and so form pastes to supplement portland cement paste, in contrast to soluble substances which act as chemical accelerants or retardants during the hydration of portland cement or otherwise modify the properties of the mixture.

For years, powdered mineral admixtures have been used in concrete to improve workability and alleviate bleeding. Materials for this purpose included cementitious materials, such as natural cement, hydrated lime, hydraulic lime, and blast-furnace slag; pozzolanic materials, such as fly ash, diatomaceous earth, clacined shale, and volcanic ash; and inert materials, such as clay, talc, and stone dust.

Although pozzolans are still used in some areas to supplement sand that is deficient in fines, air entrainment notably

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aids workability and reduces bleeding more uniformly and economically than do most of the mineral admixtures. Air entrainment also has a significant effect on increasing resistance to deterioration of concrete caused by freezing and thawing and has led to an almost complete elimination of the use of chemically inert mineral admixtures, since they do not generally impart any benefits to concrete other than improved workability and reduced segregation.

The types of mineral admixtures used at the present time are generally limited to those having pozzolanic properties. Although the major portion of this paper is devoted to a discussion of properties, methods of testing, and significance of specifications requirements for mineral admixtures having pozzolanic properties, a brief summary of the history and use of other mineral admixtures is presented.

ADMIXTURES WITH LOW REACTIVITY

This type of admixture includes such materials as ordinary clay, ground quartz, ground limestone, bentonite, hydrated lime, and talc. These materials were primarily used to improve workability prior to the advent of the use of air-entraining agents. Their value is based upon their ability to form soft, plastic pastes which separate and lubricate the aggregate particles in the fresh mortar or concrete mixture. Increasing the ratio of surface area of solids to volume of water in the paste provides an effective method of reducing the degree of bleeding. This generally increases the stiffness of the paste and at a given slump effects a wider separation of the aggregate particles in the concrete, thereby increasing the workability. The ratio of surface area of solids to volume of water may be increased by increasing the cement content or by adding a suitable mineral admixture.

The "inert" mineral admixtures vary in their ability to increase workability and reduce bleeding. Good admixtures will form pastes which contain a maximum proportion of solid matter and a minimum proportion of water. This requires that the mineral particles do not have unfavorable shapes and that the surface area is not too great. Williams [*1*][2] compared the workability afforded by several admixtures and attempted to gage the effect of each in resisting segregation in concrete. He concluded that the relative efficiency of admixtures was indicated by the volume of paste produced by a given weight of each.

CEMENTITIOUS MATERIALS

Cementitious materials used as admixtures include natural cements, hydraulic limes, slag cements, and pulverized granulated blast-furnace slag. Some of these materials, although not used as admixtures to a great extent at the present time, may provide economical means of improving specific properties of concrete.

Natural Cements:

The development of natural cements in a logical manner was started in Europe during the late 18th century. Natural cement is the product obtained by finely pulverizing calcined argillaceous limestone (ASTM Specification for Natural Cement (C 10)). Natural cements are calcined at a temperature below 1000 C, which is sufficient to drive off carbonic acid gas. This temperature is below the temperature required for the formation of tricalcium silicate and tricalcium aluminate in portland cement. However, the intimately associated compounds of calcium and silicon found in natural cement rock will react at these lower temperatures to form dicalcium silicate, the major compound in natural cement, and

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

monocalcium silicate. The percentages of these two compounds present in natural cements are dependent upon the quantity of lime present in the raw material. Because of its compound composition, natural cement produces concrete which develops strength slower than portland cement concrete. However, concretes containing natural cement show a greater rate of strength gain at later ages, greater resistance to sulfate attack, and lower heat of hydration than do concretes containing Type I portland cement. When natural cement is used as an admixture in portland cement concrete, there is no significant interaction between the hydration products of the portland cement and the natural cement. Natural cement-portland cement combinations simply alter the percentages of the hydraulic compounds present, resulting in the changes in properties of concrete mentioned.

Blast-Furnace Slag:

Granulated blast-furnace slag is the nonmetallic, amorphous product consisting essentially of silicates and aluminosilicates of calcium which is developed simultaneously with iron in a blast furnace and is produced by rapidly chilling or quenching the molten material in water, steam, or air (ASTM Specification for Portland Blast-Furnace Cement (C 205 – 64 T)). The use of blast-furnace slag as a cementitous material dates at least from 1774 when Loriot made a mortar using finely ground blast-furnace slag and slaked lime. Portland blast-furnace slag cements have been widely used in Europe since about 1855. The use of blast-furnace slags as cementitious materials is generally limited at the present time to the manufacture of portland blast-furnace slag cements and slag cements. Blast-furnace slag, for use in the manufacture of portland blast-furnace slag cement, is required by Federal Specification SS-C-197b [*2*] to comply with the following requirement for composition:

$$\frac{CaO + MgO + \frac{1}{3}Al_2O_3}{SiO_2 + \frac{2}{3}Al_2O_3} \geqq 1.0$$

Granulated blast-furnace slag having a chemical composition within the following ranges will generally meet the requirements of the above formula:

Silicon dioxide (SiO_2)	30 to 40 per cent
Aluminum oxide (Al_2O_3)	8 to 18 per cent
Ferrous oxide (FeO)	0 to 1 per cent
Calcium oxide (CaO)	40 to 50 per cent
Magnesium oxide (MgO)	0 to 8 per cent
Manganic oxide (Mn_2O_3)	0 to 2 per cent
Sulfide sulfur (S)	0 to 2 per cent

The use of granulated blast-furnace slag as a constituent of portland blast-furnace slag cement does not conform to the requirements of an "admixture," which was previously defined as ". . . materials added to the batch immediately before or during mixing." Although in practice blast-furnace slags are generally not used as admixtures, their use as such would give results similar to those obtained by intergrinding with portland cement. In addition, closer control of the desired properties of the concrete would result from the regulated additions at the mixer. Blast-furnace slags have both cementitious and pozzolanic properties. The use of blast-furnace slag in combination with portland cement in concrete results in a reduction in the total percentage of uncombined calcium hydroxide present and an increase in the quantities of calcium silicates and aluminates as compared with a corresponding concrete containing straight portland cement. Such concrete will have a lower temperature rise, may tend to inhibit expansion due to alkali-aggregate reaction, will likely exhibit decreased permeability, and may exhibit increased strength at later ages and increased resistance to aggressive attack of sea water, sulfate-bearing soil solutions, and natural acid waters, as compared to concrete containing a Type I portland cement. It should be noted that these advantages will be gained only under suitable curing conditions for the concrete and may vary greatly with the composition and fineness of the slag and the composition and fineness of the cement. The relative improvements are likely to be greater for concrete of low cement content.

Slag Cement:

Slag cement is defined under ASTM Specification for Slag Cement (C 358 – 64 T) as "the finely divided material consisting essentially of water-quenched granulated blast-furnace slag and hydrated lime. It shall consist of at least 60 per cent water-quenched blast furnace slag by weight." Slag cements have lower strengths than portland blast-furnace slag cements. Because of their low strength, they are generally used in combination with portland cement in concrete. In the United States, the proportions usually used are about 50 per cent slag cement and 50 per cent portland cement. Economy is the primary advantage to be derived from the use of slag cement. Concrete containing slag cement in combination with portland cement gains strength slower and may have less temperature rise than corresponding straight portland cement concrete. At later ages, however, the strengths of slag cement-portland cement concrete which has been moist cured are generally comparable to straight portland cement concrete. Concrete containing this type of cement usually exhibits good resistance to sulfate attack and better plasticity than does corresponding straight portland cement concrete. However, most countries have abandoned production of this cement, because it is relatively unstable in storage and is lower in strength-producing characteristics.

ASTM Specification (C 358) and Federal specifications [*3*] cover two types of slag cements, Types S and SA. Type S is defined as slag cement for use as a blend with portland cement in making concrete and as a blend with hydrated lime in making masonry mortar. Type SA is an air-entraining slag cement for the same uses as Type S. Chemical requirements for slag cements are the same for both types. These limits require the composition of the slag to conform to the following:

$$\frac{CaO + MgO + \frac{1}{3}Al_2O_3}{SiO_2 + \frac{2}{3}Al_2O_3} \geqq 1.0$$

The chemical requirements for the slag cement itself are directed toward limiting the quantities of certain compounds present. The chemical requirements listed in ASTM Specification C 358 are as follows:

Sulfur trioxide (SO_3), max	4.0 per cent
Sulfide sulfur (S), max	2.0 per cent
Insoluble residue, max	1.0 per cent
Loss on ignition, max	4.0 per cent

Specification C 358 also has limitations on the types and quantities of processing additions which may be interground with slag cements. These limits are as follows:

Material	Per Cent by Weight of Cement
TDA	0.043
109B	0.03
Polyfon T	0.05
S-TDA	0.083

Federal Specification SS-C-218a contains the chemical requirements listed above and, in addition, has a limitation on manganic oxide (Mn_2O_3) of not more than 1.5 per cent.

The physical test limits for slag cements require that the specific surface by the Blaine air permeability method be an average of not less than 4700 cm^2/g with no single value being less than

4200 cm^2/g. This fineness which is approximately 50 per cent higher than that found in most portland cements, helps to improve the strength-producing properties of the slag. The soundness, time of setting, and air content of mortar requirements for slag cements are similar to those for portland cements. The compressive strength requirements for Type S slag cement at 7 and 28 days' age are 600 and 1500 psi, respectively.

Hydraulic Lime:

Hydraulic lime is obtained from the burning of limestone containing clay. The alumina and silica of the clay combine in some degree with the lime produced in the burning, and the resulting compounds are responsible for the hydraulic properties of the material. The composition of limes may vary from high quality limes, containing less than 1 to 2 per cent silica and alumina, to hydraulic limes containing up to 25 per cent of these constituents. As the content of silica and alumina increases, the rapidity of slaking and the evolution of heat, which is characteristic of limes high in CaO decreases until, with the limes having a high degree of hydraulic activity, no appreciable immediate reaction occurs unless they are finely ground. Hydrated limes, both with and without hydraulic properties, have been used in the past as admixtures for concrete.

ASTM Specification for Hydraulic Hydrated Lime for Structural Purposes (C 141) requires that calcium and magnesium oxides comprise between 60 and 70 per cent of the hydraulic hydrated lime. The silica content, which, along with iron and aluminum oxides, combines with the calcium and magnesium oxides and imparts the hydraulic properties to the lime, is required to be between 16 and 26 per cent. The total of iron and aluminum oxides is limited to a maximum of 12 per cent, and carbon dioxide may not exceed 5 per cent. In addition to the chemical requirements, the lime must also conform to physical requirements for fineness, time of setting, soundness, and compressive strength. Requirements for fineness are that not more than 0.5 per cent may be retained on a No. 30 sieve and not more than 10 per cent may be retained on a No. 200 sieve; time of setting limitations are a maximum of 2 hr for initial set, and 48 hr for final set. The autoclave test for soundness permits not more than 1.0 per cent expansion; the compressive strength requirements are a minimum of 175 psi at 7 days' age and 350 psi at 28 days' age.

At the present time, hydraulic hydrated lime is rarely used as an admixture in concrete, primarily due to the lack of any real advantage being derived from such use. One of its principal uses is as an ingredient in masonry mortars.

POZZOLANIC MATERIALS

The term "pozzolan" is employed to designate a siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties (ASTM Definition of Terms Relating to Hydraulic Cement (C 219)). Calcium hydroxide is liberated during the hydration of portland cement, and pozzolans combine with this liberated calcium hydroxide to form stable cementitious compounds which contribute to strength and watertightness. It is generally agreed that the siliceous ingredient of a pozzolan should be in an amorphous or noncrystalline state, such as glass, opal, or thermally altered clay for best activity. Crystalline siliceous materials, such as quartz even in finely divided form, combine, but slowly, with lime, except with curing at elevated temperatures, and, therefore, are considered to be quite inactive.

The early Greeks and Romans used pozzolans in combination with lime to improve the cementing qualities of lime. Such materials were used in a variety of constructions, including the aqueducts of ancient Rome, some of which are still in service. The first large scale use of pozzolan in the United States took place in 1910 when a rhyolite tuff was used in the construction of the Los Angeles aqueduct. This was followed by the use of ground granite in Arrowrock Dam and pulverized sandstone in Elephant Butte Dam, which were built by the Bureau of Reclamation between 1911 and 1916. Although the degree of pozzolanic activity of the Arrowrock ground granite and Elephant Butte ground sandstone was very low, especially considering modern specifications requirements, they were interground with cement to produce "sand cements," which produced strengths in tension tests equal to straight portland cement at one year and gave greater strengths at five years' age. The use of varying amounts of pozzolan in concrete, particularly in mass concrete construction, has continued intermittently since that time.

TABLE 1—CHEMICAL REQUIREMENTS FOR POZZOLANS.

	Bureau of Reclamation [4]		Corps of Engineers [5]		ASTM	
	Natural Pozzolan	Fly Ash	Natural Pozzolan	Fly Ash	Natural Pozzolan (C 402)	Fly Ash (C 350)
Silicon dioxide (SiO_2) + aluminum oxide (Al_2O_3) + ferric oxide (Fe_2O_3) not less than..............................	75.0	75.0	70.0	70.0	70.0	70.0
Magnesium oxide (MgO), not more than..	5.0	5.0	5.0	5.0	5.0	...
Sulfur trioxide (SO_3), not more than.....	4.0	4.0	4.0	4.0	3.0	5.0
Loss on ignition, not more than........	10.0	5.0	10.0	6.0	10.0	12.0
Moisture content, not more than.......	3.0	3.0	3.0	3.0	3.0	3.0
Exchangeable alkalies as Na_2O, not more than............................	2.0	2.0	1.5	1.5	...	1.5

The amounts of pozzolan used in concrete has varied from 5 per cent or less of the total cementitious material, when employed solely for the purpose of improving workability, to between 15 and 40 per cent, when utilized for its pozzolanic properties. In the United States, the amount used generally ranges between about 20 to 35 per cent of the total cementitious material. In Italy, where pozzolans interground with portland cement are used extensively, an attempt is made to proportion the amounts used to obtain a balance between the total amount of silica, alumina, and iron oxide with the total amount of calcium oxide available in the cement-pozzolan mixture. In this way, most of the free lime in cements is combined with silicates and aluminates, the cementitious properties being thereby more completely utilized. To conform to this principle, Italian specifications require that the lowest quantity of pozzolan to be used with a cement be determined by the following composition ratio to which the cement-pozzolan mixture in total should agree:

$$\frac{SiO_2\,\% + Al_2O_3\,\% + Fe_2O_3\,\%}{CaO\,\%} \geq 1$$

Pozzolans are classified into two major groups: (1) raw or calcined natural and (2) artificial. Natural pozzolans include such materials as some diatomaceous earths, opaline cherts and shales, tuffs, and volcanic ashes or pumicites. Each may or may not require calcination, depending upon its clay content or grinding or both in order to be suitable for use as pozzolan. Artificial pozzolans include the fine fly ash produced from the burning of powdered coal which is caught in electrostatic precipitators, water-quenched boiler slag, and precipitated silica. Suitable fly ash has a low-carbon content and a fineness usually about the same as that of portland cement. Most of the fly ash particles are in the form of tiny glassy spheres; usually the low carbon fly ashes of the United States require no further processing. The spherical shape and texture of the low carbon fly ashes generally result in a reduced water requirement when used in concrete. On the other hand, concrete containing most natural or calcined natural pozzolans requires more water than does the corresponding portland cement concrete.

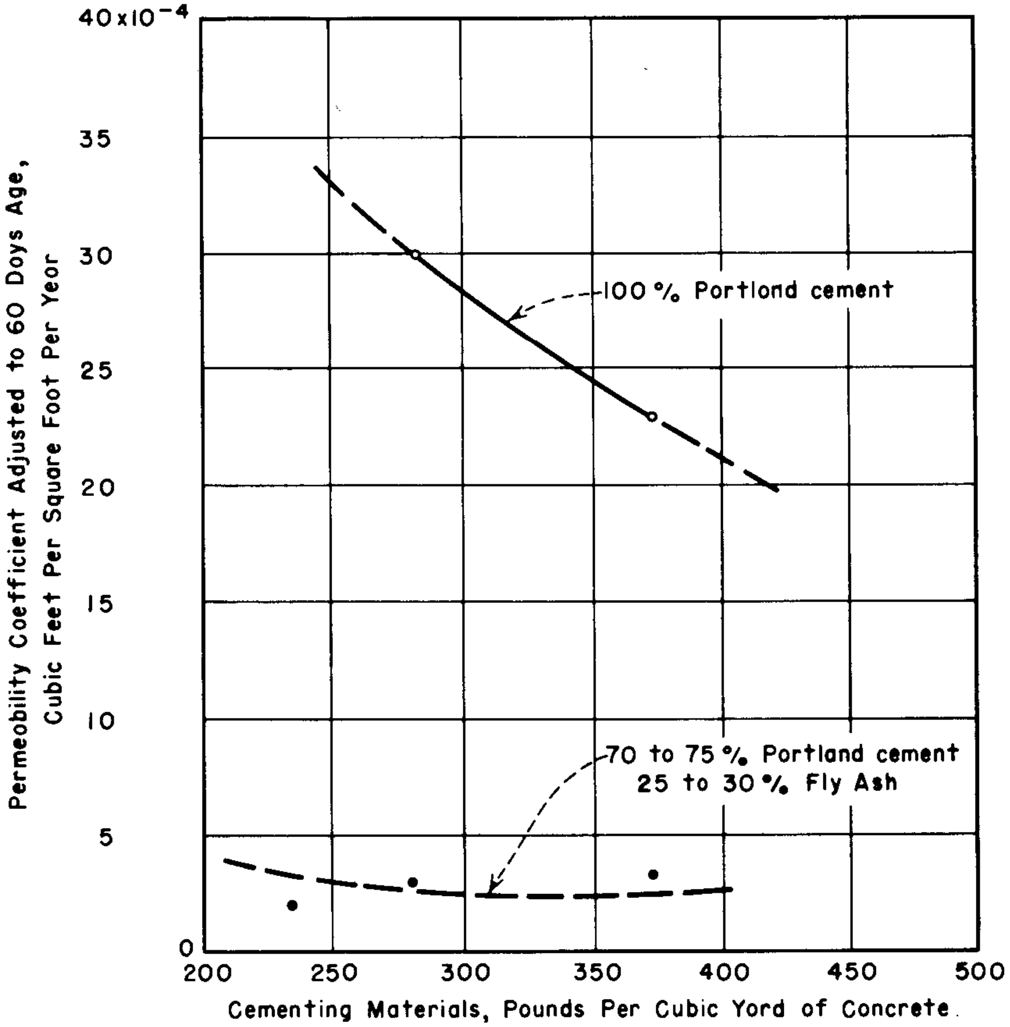

FIG. 1.—Permeability of concrete with and without pozzolan.

The American Society for Testing and Materials, the Bureau of Reclamation, and the Corps of Engineers have all established specifications for pozzolan. Chemical limitations, in per cent, imposed by each of the three specifications are tabulated in Table 1.

These requirements provide for a minimum of 70 to 75 per cent of the pozzolan to be composed of silicon dioxide, aluminum oxide, and ferric oxide. The other main compounds present in pozzolans have maximum limits placed upon the quantities permitted to be present. These compounds are magnesium oxide, sulfur trioxide, loss on ignition, moisture content, and exchangeable alkalies.

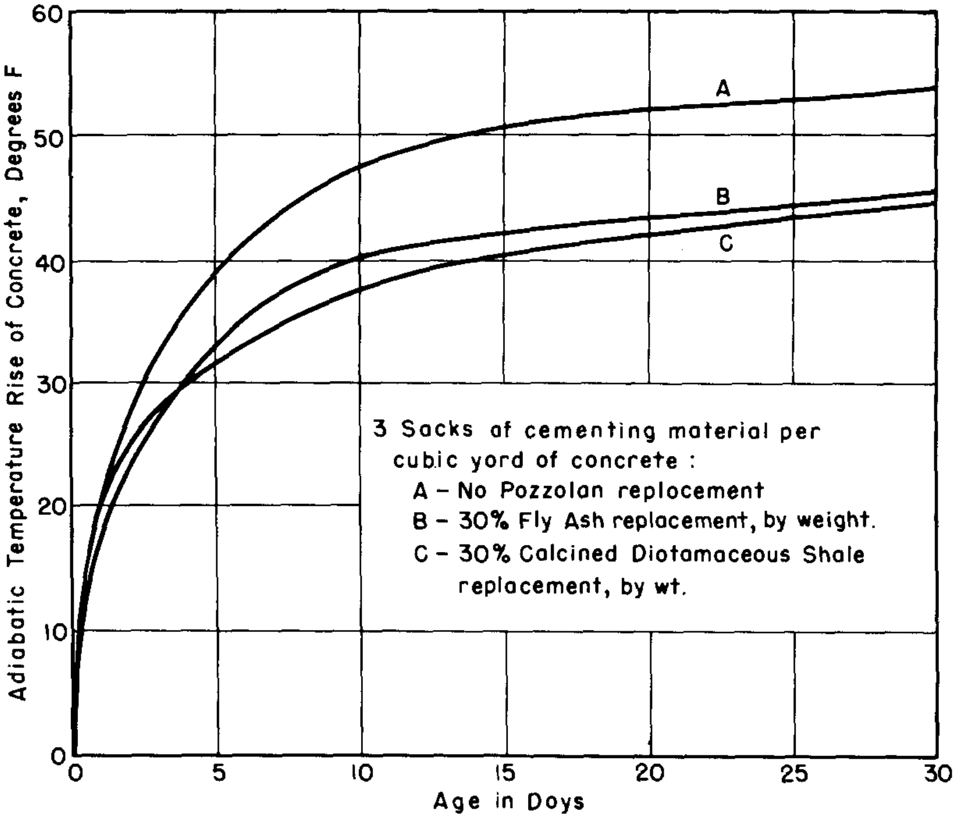

FIG. 2.—Effect of pozzolan on temperature rise of concrete.

The principal technical benefits generally derived from the use of pozzolan in concrete are increased impermeability (Fig. 1) [*6*], lower heat of hydration (Fig. 2) [*6*], reduced alkali-aggregate expansion, and improved workability. One of the disadvantages of using pozzolan is that concrete containing pozzolan generally develops strength more slowly than portland cement concrete. However, concretes containing some pozzolans under moist or mass curing conditions continue to gain in strength for a long period of time and may produce concretes whose strengths are equal to or substantially greater than the strength of corresponding straight portland cement concretes. In mass concrete, this type of strength gain is satisfactory. However, paving and structural concrete generally require early strength properties comparable to those obtained in straight portland cement concrete. Therefore, when pozzolan is used for these purposes, it is necessary to proportion the concrete mixes to provide higher early strengths.

One method frequently used in certain localities to partially counteract the lower strength of portland cement-pozzolan concrete at early ages is the redesign of concrete mixtures to incorporate a greater amount of pozzolan in the mix than the cement it replaces [*7,8*]. Redesign of mixtures in this manner can be justified economically only in localities where the cost of the pozzolan is low enough to permit production of this concrete without an increase in cost. Pozzolans lower the resistance of concrete to deterioration caused by freezing and thawing unless longer than usual moist curing is provided, which in most instances is not practicable. The magnitude of the reduction is usually not large and varies with the pozzolan. However, under prolonged moist curing conditions, many pozzolans have a beneficial effect on the durability of concrete as measured by freezing and thawing tests.

TABLE 2—PHYSICAL REQUIREMENTS FOR POZZOLANS.

	Bureau of Reclamation		Corps of Engineers		ASTM	
	Natural Pozzolan	Fly Ash	Natural Pozzolan	Fly Ash	Natural Pozzolan	Fly Ash
Fineness						
Mean particle diameter, microns, not more than.....	...	...	...	...	9.00	9.0
Specific surface, Blaine air permeability apparatus, cm²/cc, not less than.....	16 500	7 000	6 500	6 500	...	...
Material retained on No. 325 mesh sieve, per cent, not more than..............	12	12	...	...	12.0	...
Compressive strength						
With portland cement, per cent of control, 28 days, not less than............	80	80	75	75	75	85
With lime, 7 days, min psi..	800	800	900	900	600	800
Increase in drying shrinkage of mortar bar, per cent shrinkage of pozzolan bar minus per cent shrinkage of control bar, not more than...................	0.04	0.04	0.03	...	0.03	0.03
Water requirement, per cent, not more than..........	112	103	0[a]	0[a]	115	105
Reduction of reactive expansion at 14 days, per cent, not less than............	60	60	75	75	75	...
Mortar expansion at 14 days, per cent, not more than...	...	...	...	...	0.020	0.020
Soundness, autoclave expansion, per cent, not more than...................	...	...	0.50	0.50	0.50	0.50

[a] This specification requires that a mortar with a natural pozzolan and 115 per cent of the water content of the control mortar or a mortar with a fly ash pozzolan and 103 per cent of the water content of the control shall have a flow equal to or greater than that of the control.

Conformance with the specifications limits listed in Table 2 generally will assure that pozzolan will impart the desired properties to concrete in which it is used.

The fineness requirement is important to assure that a uniform and sufficient surface area per unit quantity of material is present to react with the calcium hydroxide which is liberated during the hydration of cement. The fineness of a pozzolan will affect its other properties, primarily its strength-producing ability. The more finely ground a material is, the more rapid is the rate of chemical reaction and the greater is the proportion of the pozzolan which reacts. The workability of concrete is decreased as the fineness is decreased. Coarse pozzolans of poor particle shape, particularly volcanic glasses, require a larger amount of water in concrete for a given slump. The water requirement of concrete may also be increased by pozzolans of very high fineness, such as some diatomites, when used in large amounts. The greater the water requirement of a pozzolan, the greater will be the drying shrinkage of the concrete in which it is used. Coarse pozzolans of poor particle shape, such as some of the coarse volcanic ashes, also contribute to bleeding in freshly mixed concrete.

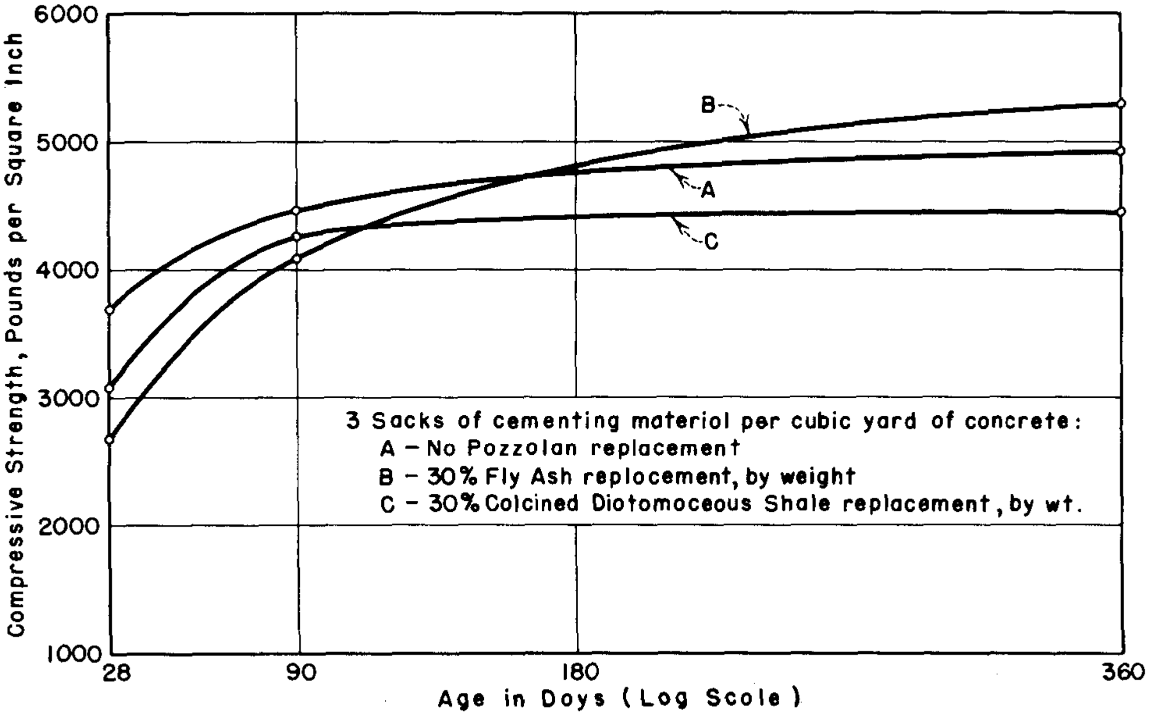

FIG. 3.—Effect of pozzolan on compressive strength of concrete.

Methods of test in use for determination of fineness of pozzolans include specific surface (cm^2/g or cm^2/cc of solid volume) by Blaine air permeability apparatus, per cent of material retained on a No. 325 mesh sieve, and calculated mean particle diameter.

The compressive strength of portland cement-pozzolan mortar is a property of considerable importance, since the value of a pozzolan when employed in combination with portland cement depends primarily upon its effect upon mechanical strength of concrete. The contribution of a pozzolan to strength of concrete arises in the cohesion of the hydration products of cement-pozzolan combinations and in the adhesion of the matrix to the grains of sand or other aggregate. Concretes containing pozzolan, when fog-cured 28 days, usually produce strengths which are lower than similarly cured concrete containing the same weight of portland cement only. However, the difference in strength becomes progressively less as the age of the concrete increases, and, at later ages, concrete containing optimum amounts of pozzolan frequently develops higher strengths (Fig. 3) [*6*]. The slower strength-producing properties of portland cement-pozzolan concretes are considered to be due to the slow rate of reaction between the calcium hydroxide liberated as the cement hydrates and amorphous silica or siliceous glass to form silicates and aluminosilicates of lime that are not found in portland cement concretes which do not contain pozzolan. An evaluation of the compressive strength-producing properties of pozzolans is made by comparing the compressive strengths of portland cement-pozzolan mortars, as determined from tests on 2-in.-cube specimens, with compressive strengths of a similar mortar containing the same portland cement without pozzolan.

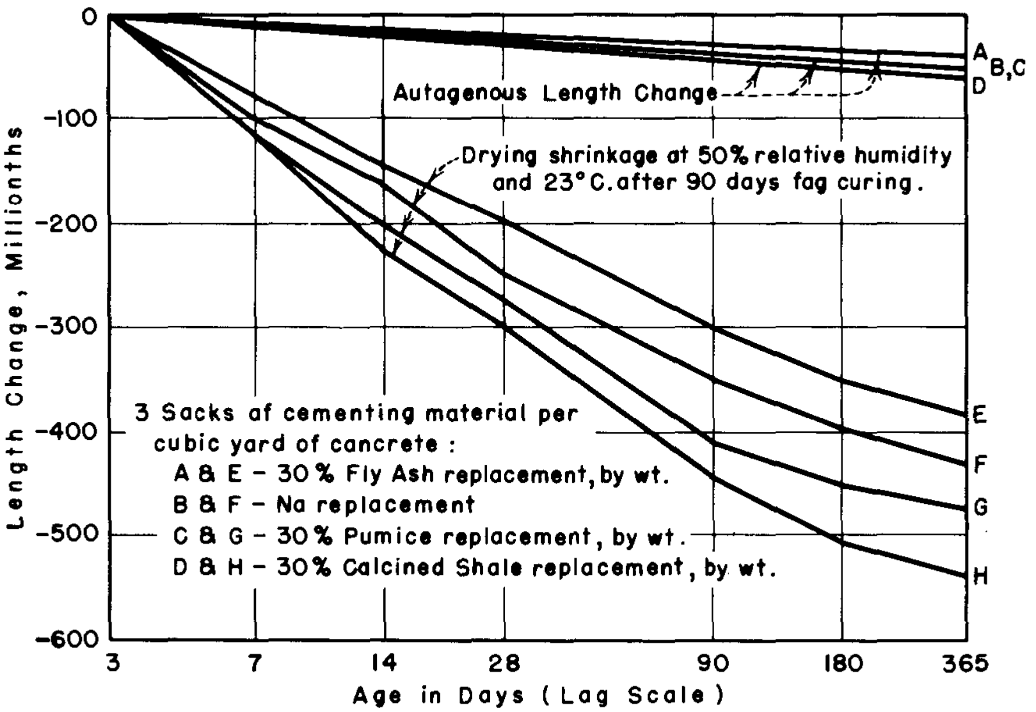

FIG. 4—Drying shrinkage and autogenous length change of 4 by 4 by 30-in. bars of portland cement concrete with various pozzolan replacements.

Another test used for determining the strength-producing properties of pozzolan is the test for compressive strength with lime, which test is considered effective in determining the total activity of pozzolan (ASTM Specification for Fly Ash for Use as a Pozzolanic Material With Lime (C 379 – 65 T)). Pozzolan will react with hydrated lime in the presence of moisture developing a "set" by the formation of cementitious products and increasing in strength to an extent that depends on the activity of the pozzolan. Therefore, a lime-pozzolan-sand mortar is used to obtain an evaluation of the activity of the pozzolan without introducing other variables from portland cement. Results of this test are generally reported after 7 days' age and, therefore, provide a more rapid evaluation of the strength-producing properties of a pozzolan than do portland cement-pozzolan strengths which are generally reported at 28 days' age. It is considered that the portland cement-pozzolan mortar test provides a more reliable indication of the actual performance to be expected from concrete containing a given pozzolan. However, the lime-pozzolan test for compressive strength is also useful as a uniformity test for compressive strength after a source has been established.

As previously mentioned, a direct relationship exists between water requirement and drying shrinkage; the greater the amount of water required, the greater the drying shrinkage. When pozzolan is added to concrete as a partial cement replacement, the water requirement for concrete of a given consistency is usually changed. Natural pozzolans have a tendency to increase the water requirement, and fly ashes of low carbon content usually lower it slightly. A high-water requirement will increase drying shrinkage of concrete, which in turn will increase the amount of cracking which may occur, and may harmfully affect other properties of concrete, such as strength, durability as measured by freezing and thawing, impermeability, and sulfate resistance. Pozzolan specifications limit the per cent increase in both water requirement and drying shrinkage of mortar containing portland cement-pozzolan combinations over that of portland cement mortar without pozzolan, to assure that the shrinkage characteristics of concrete containing pozzolan are not adversely affected to a harmful degree. Figure 4 [*6*] shows the effect of various pozzolan replacements on drying shrinkage and autogenous length change of portland cement concrete.

One of the important benefits derived from the use of pozzolan is its effectiveness in controlling potentially disruptive expansion in concrete resulting from the formation of an alkali-silicate gel, which is the product of reaction between certain types of mineral aggregates (cherts, opal, and so on) and the alkalies (sodium and potassium oxide) in cement.

Initially, the only apparent methods of controlling alkali-aggregate reaction in concrete were to use low-alkali cements or to avoid the use of reactive aggregates. Subsequently, some pozzolans were found to effectively reduce expansion caused by alkali-aggregate reaction. Other pozzolans have little effect in controlling this expansion. It is, therefore, necessary to test pozzolans individually in order to evaluate their ability to control this expansive reaction. An accelerated test, which measures the effectiveness of a pozzolan in reducing expansion of mortar made with high-alkali cement and reactive aggregate, is generally used for this purpose. The amount of pozzolan needed in concrete to control reactive expansion will vary with the individual pozzolan, aggregates, and with the alkali content of the cement.

ACI Committee 212 [*9*] reports the following on alkali-aggregate reaction:

> . . . Ample protection should generally be obtained by use of proportions (of pozzolan) ranging from 20 to 35 per cent by weight of the cement. The use of these amounts usually does not impair physical properties of the concrete such as strength and workability. Certain materials, however, when finely divided and of high opal content (e.g., certain diatomaceous earths and opaline cherts) will prevent expansion when used in amounts of less than 15 per cent by weight of the cement. In proportions of 10 per cent or less by weight of the cement, certain pozzolans may increase expansion of concrete containing reactive aggregate and high-alkali cement, presumably because interaction of a portion of the cement alkalies with the pozzolan produces a ratio of reactive silica to the available alkalies which more closely approaches the optimum (or 'pessimum') for formation of expansive alkali-silica gel.

As a safety precaution in concrete construction against the possible increased expansion of concretes containing small amounts of certain pozzolans, some organizations specify that pozzolans should not be used in amounts less than 15 per cent by weight of total cementitious material.

The potential danger of unsoundness occurring in concrete due to the presence in pozzolan of either the periclase form of magnesia, "dead burnt" calcium oxide, or an excessive amount of sulfur trioxide, points out the importance of determining the characteristics of the pozzolan in this regard. The expansion of mortar bars determined by the autoclave soundness test, as required for portland cements, is the procedure used for evaluation of the effect of a pozzolan on soundness of concrete.

Much has been done in recent years toward the development and refinement of the use of mineral admixtures. Through improved technology and a thorough knowledge of proper design of concrete mixes to meet specific needs, the use of many admixtures previously used has been eliminated. In addition, this improved technology has resulted in more economical and advantageous uses of mineral admixtures, which have provided better concrete at lower cost. Based upon present technological advances, it is anticipated that the future will continue to show marked improvements in concrete technology, which will undoubtedly include further refinements in the use of mineral admixtures.

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STP169A-EB/Apr. 1966

Other Materials

CHEMICAL ADMIXTURES

BY BRUCE FOSTER,[1] Personal Member, ASTM

Although concrete admixtures are chemicals in a literal sense, by convention in concrete technology, the term "chemical admixture" is restricted to water-soluble substances other than air-entraining agents. Most chemical admixtures react chemically with the cement in concrete. This discussion will be confined to set-controlling and water-reducing admixtures. Chemical admixtures of other types are classified and described in a recent report of the American Concrete Inst. [*1*].[2]

A water-reducing admixture is defined as one that reduces the quantity of mixing water required to produce concrete of a given consistency; a retarding admixture as one that retards the setting of concrete; an accelerating admixture as one that accelerates the setting and early strength development of concrete; a water-reducing and retarding admixture as one that reduces the quantity of mixing water required to produce concrete of a given consistency and retards the setting of concrete; and a water-reducing and accelerating admixture as one that reduces the quantity of mixing water required to produce concrete of a given consistency and accelerates the setting and early strength development of concrete. These five types of admixtures are referred to in ASTM Specifications for Chemical Admixtures for Concrete (C 494 – 63 T) as Types A, B, C, D, and E, respectively.

TYPES OF MATERIALS AND THEIR ACTION IN CONCRETE

Many materials, when incorporated in small amounts into concrete have been found to modify the setting properties of portland cement [*1–3*]. Often, use of such materials also results in a lower water requirement to produce a given slump.

The materials in wide-spread use as set-retarding and water-reducing admixtures in the United States were classified in the 1959 *ASTM Symposium on Effect of Water-Reducing Admixtures and Set-Retarding Admixtures on Properties of Concrete* [*4*] into four classes: (1) lignosulfonic acids and their salts, (2) modifications and derivatives of lignosulfonic acids and their salts, (3) hydroxylated carboxylic acids and their salts, and (4) modifications and derivatives of hydroxylated carboxylic acids and their salts. In each of these, the primary component has both water-reducing and set-retarding properties. In the formulation of products of Classes 2 and 4 these admixtures may be modified by the addition of other components to give various degrees of retardation, no significant change in setting time, or acceleration, while at the same time preserving the water-reducing properties. They also may be modified by the addition of an air-entraining admixture.

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

The accelerating admixture which has found greatest use is calcium chloride [*1,5*]. The organic material triethanolamine is also used extensively, but usually in combination with other materials in a proprietary mixture, and little published information is available on its properties [*6–8*]. The discussion on accelerators here, therefore, will be confined primarily to the properties of calcium chloride.

The mechanisms whereby gypsum and the alkalies in cement affect its setting time are not fully established, nor are those by which retarders and accelerators still further modify the setting. It is to be expected that the extent of any modification will depend upon the properties of the cement to which the admixtures are added. The effect of calcium chloride on acceleration of strength development has been reported to vary with the cement with which it is used [*3*], while the behavior of concrete containing water-reducing retarders appears to be dependent even more upon the properties of the cement. Polivka and Klein [*9*] reported that the effectiveness of the latter materials, both from a water-reducing standpoint and from a retarding standpoint, was greater with cements of low alkali and low C_3A content. Tuthill, Adams, Bailey, and Smith [*10*], Palmer [*11*], and Tremper [*12*] have reported excess retardation when retarders were used with certain cements, which could be overcome by an increase in the sulfate content of the cement. Often, the excess retardation was accompanied by stiffening, even though the cement without the water-reducing retarder showed no early stiffening and had a normal setting time. Seligman and Greening have reported on work which provides information on the chemistry of this phenomenon [*13*].

Bruere [*14*], and Dodson and Farkas [*15*] have noted that the effect of water-reducing retarders depends in some cases on the time at which the retarder is added to the mixture. Dodson and Farkas, working with cements little affected by the addition of either lignosulfonate or hydroxylated carboxylic admixtures, found that the efficiency of the admixtures as retarders, and their capacity to enhance air entrainment and reduce water requirement, was greatly increased by a 2-min delay in their addition after mixing was commenced.

The unfavorable behavior with certain cements and certain conditions of use which have been reported are counterbalanced by a record of successful use under controlled conditions in a substantial portion of present day concreting operations. However, they indicate that where experience with specific admixture-cement combinations under similar job conditions is not available, tests with job combinations should precede construction.

Where two or more admixtures are added to the concrete mixture, they should be added separately during the mixing operation unless they have been shown to be compatible when added to the concrete as a single material.

EFFECTS ON PLASTIC CONCRETE

Water Reduction:

The water reduction resulting from the use of water-reducing admixtures ranges from 5 to 15 per cent [*16*]. The water reduction found with lignosulfonate water-reducers may be partly brought about by the air entrained by these materials. In addition to varying with the particular cement employed, the amount of water reduction with a given admixture is also influenced by dosage, cement content, type of aggregate, and the presence of other admixtures, such as air-entraining agents or pozzolans.

Water-reducing admixtures have been reported to be effective with all types of portland cement, portland blast-furnace slag cement, portland pozzolan cement, and with high alumina cement.

The addition of recommended amounts of calcium chloride has been found to reduce the water requirement by a small, but definite, amount over that required to produce the same slump, with no chloride added.

Setting Time:

The magnitude of the effect on setting time of the addition of calcium chloride depends not only upon the dosage, but also upon the particular cement, the temperature, and other factors. The recommended maximum dosage has a substantial effect on setting time at normal temperatures, and can produce, at high temperatures, a very rapid set, as is also the case with very large dosage at normal temperatures.

Similarly, the retardation of setting time brought about by retarders is dependent not only upon the particular cement with which they are used, but also upon the temperature, dosage, and other factors. Overdosage may produce excessive setting times of 24 hr or more, but in such cases, if the concrete finally sets and has been protected from drying, ultimate strengths developed may be satisfactory if forms are left in place for a sufficient length of time. With either lignosulfonate or hydroxylated carboxylic admixtures, the degree of retardation can be controlled by varying the dosage, provided that the allowable air content with lignosulfonate materials is not exceeded. Classes 2 and 4 water reducers may be formulated to give no retardation or to produce acceleration, both effects being produced by the incorporation of a catalyst or an accelerator.

Air Entrainment:

Lignosulfonate water-reducing retarders usually entrain 2 to 3 per cent of air when used in normal dosages. The hydroxylated carboxylic admixtures do not entrain air, nor does calcium chloride. However, all three materials enhance the effectiveness of air-entraining agents from the standpoint of volume of air produced, so that less air-entraining agent is required when added to concrete containing one of these other admixtures. Air-entraining properties may be incorporated in the modified materials of Classes 2 and 4 by the incorporation of a suitable air-entraining agent. When the air-entraining agent is not incorporated in another admixture by the manufacturer, it and other admixtures should be added to the concrete separately unless tests have shown that the two materials are compatible when added to the concrete as a single material.

Bleeding:

Water-reducing admixtures that entrain air reduce bleeding, the reduction being due to the entrained air and the lower water content. Hydroxylated carboxylic water-reducing retarders have been reported to increase the rate and amount of bleeding. Such bleeding has been suggested as being responsible for a portion of the strength increase observed with the use of these materials.

Slump Loss:

Contrary to expectations, water-reducing retarders have usually not been found effective in reducing slump loss resulting from substantial delays, after mixing, in placing the concrete. As pointed out above, the use of retarders with some cements may actually produce an early stiffening. However, the addition of a water-reducing admixture will give a higher initial slump with the same water-cement ratio and permit more slump loss before concrete becomes unworkable.

Calcium chloride may result in early stiffening and in many cases, therefore, is not added until after mixing has commenced.

EFFECTS ON HARDENED CONCRETE

Strength:

Usually compressive strength is increased 10 to 20 per cent by use of a water-reducing admixture based on lignosulfonate or a salt of a hydroxylated carboxylic acid [*1*]. The percentage of strength gain at 3 and 7 days is usually higher than that at 28 days, while tests up to five years in duration have shown a continued strength benefit.

As pointed out earlier, lignosulfonate water-reducers normally entrain some air. The portion of the water reduction attributable to the air content can, with suitable adjustment of the amount of sand, compensate largely or wholly for the loss of strength due to the entrained air. The balance of the water reduction when using a lignosulfonate water reducer, and the reduction produced by a hydroxylated carboxylic water reducer, are effective in increasing the 28-day strength over that which would be produced with similar concrete without admixtures. Further, this increase in strength is generally greater than would be predicted from the reduction in water requirement produced.

In common with retarders, calcium chloride also shows maximum strength gain at early ages [*17*]. Concrete with 2 per cent calcium chloride has often been found to be stronger at one year than similar concrete without the admixture. Accelerators are particularly effective at very early ages and at relatively low temperatures.

Flexural strength generally is improved by all three of these admixtures, but the effect is less pronounced than that on compressive strength, and calcium chloride may even cause some reduction in flexural strength at late ages.

Permeability:

The permeability of concrete is not changed significantly by any of the chemical materials, except that the concrete matures more rapidly, and in the case of accelerators may be affected less by failure to provide effective curing.

Drying Shrinkage:

The available data on the effect of accelerators and retarders on drying shrinkage are conflicting, probably because of the influence of variations in test procedures employed. Tremper and Spellman [*8*], using concrete specimens of 3 by 3-in. cross section made from a blend of Type II cements, moist cured for 7 days, and dried at 50 per cent relative humidity for 28 days, found drying shrinkages of 8 to 17 per cent greater than similar concrete without admixtures for one hydroxylated and two lignosulfonates, and shrinkages of 30 per cent greater for calcium chloride. The per cent increase in shrinkage over the control concrete was usually higher for shorter than for longer drying periods, particularly with accelerators. The relative shrinkage where calcium chloride was added was found to be dependent on the SO_3 content of the cement used. Other investigators [*7*,*17*–*19*], using various procedures and drying times, have reported drying shrinkage figures which are usually lower than those of Tremper and Spellman.

In assessing the importance of these figures it should be kept in mind that they are comparable in magnitude with effects which may be introduced through choice of cement, choice of maximum aggregate size, contamination of aggregate, and choice of aggregate source.

Resistance to Freezing and Thawing:

While the entrained-air void spacing obtained with Class 1 water-reducing retarders is slightly greater than that for an equivalent amount of entrained air produced by the widely used air-entraining agents, the performance of concrete containing water-reducing retarders, as measured by freezing and thawing tests, has often been found to be better than concrete of the same air content, but without the water-reducing retarders. This increase might be the result of the reduction in water-cement ratio.

Calcium chloride has been found to improve the early resistance of concrete to freezing and thawing, either in the presence or absence of ice-removal salts, but to reduce somewhat the eventual durability of the fully cured concrete [*17*, *18*,*20*].

Resistance to Sulfates:

Laboratory tests have shown some small improvement in sulfate resistance, through the use of water-reducing retarders [*18*], but the use of calcium chloride has been found to decrease somewhat the resistance of concrete to sulfates [*17*].

Heat of Hydration:

The chemical admixtures have been shown to have little, if any, effect on the total heat liberated during the hydration of cement [*6*,*18*], but they may have a pronounced effect upon the rate at which the heat is liberated. Concrete containing calcium chloride liberates heat earlier, as would be expected from the earlier strength development.

Corrosion of Metals:

The addition of calcium chloride to reinforced concrete has not been found to contribute significantly to corrosion of the reinforcing. If the concrete is adequately designed, properly consolidated to form a continuous contact with the steel, and of adequate cover thickness, no corrosion problems are normally encountered. However, chlorides should not be used in prestressed concrete [*21*,*23*]; in concrete where stray currents are present [*24*]; in concrete in which dissimilar metals are imbedded, such as aluminum conduit and steel reinforcement [*25*]; or where galvanized forms are to be left in place [*26*]. Also calcium chloride may bring about corrosion where elevated temperature is employed during curing [*27*]. By contrast, another accelerator, stannous chloride [*27*], when properly used, was found not to contribute to corrosion. The usual water-reducing retarders, unless modified by the addition of chlorides, have not been found to bring about corrosion. There is some evidence that retarding admixtures, when used in conjunction with chlorides, reduce the increased electrical conductivity which would normally result from use of the chlorides, and hence the resulting corrosion [*24*].

APPLICATIONS

Water-Reducing Admixtures:

Water-reducing admixtures may be used with no change in cement content and slump to produce a lower water-cement ratio; with no change in cement content and water-cement ratio to produce a higher slump; or with reduced cement content and unchanged water-cement ratio and slump. In the first case the usual benefits accruing from the use of a lower water-cement ratio will normally be obtained, and as pointed out above, in many cases an increase in strength greater than normally produced by the reduction in water possibly may result. In the second application easier placing of concrete, or a higher slump with delayed placing of the concrete, may be obtained. In the third application monetary economies may result.

Retarders:

Retarders may be used to delay the setting of concrete during hot weather, or to extend the vibration limit so that large members can be cast and consolidated without cold joints, and without damage to the freshly placed concrete due to settlement of forms as concreting proceeds.

Accelerators:

Accelerators have their primary application in cold weather concreting where they may be used to expedite the start of finishing operations, and in certain cases, the application of insulation; reduce the time required for proper curing; and permit earlier removal of forms or loading of the concrete.

Dosage:

In the case of water-reducing and retarding admixtures dosages required to produce specific results are usually recommended by the manufacturers. Variation in the dosage can often be made to obtain the desired concrete properties under particular job conditions. In other cases, the manufacturer may change the formulation of the admixture to suit the conditions under which it will be used. Increase in the dosage in a multipurpose material to obtain one particular effect might not be feasible because, as an example, too much or too little air-entrainment might result.

Calcium chloride is usually added in amounts of 1 to 2 per cent by weight of the cement, and the latter figure should not be exceeded.

SPECIFICATIONS

A specification for a concrete admixture should serve several purposes.

1. It should provide test methods and specification limits by which the material to be tested may be judged as to its general ability to perform the functions for which it is purchased. Tests for strength, water requirement, and setting time measure properties important in the use of chemical admixtures.
2. Specifications should provide test procedures and test limits against which the material may be judged from the standpoint of not producing deleterious properties in the concrete. Excessive shrinkage, low resistance to freezing and thawing, and excessive setting time are examples of such properties.
3. The specification should provide procedures whereby the performance of a particular admixture with particular sources of cement and aggregate, and specific job conditions can be assessed.
4. A specification should provide a ready means of identifying materials in successive shipments, both from the standpoint of composition and concentration, to give the user some assurance that the material being used is uniform and is the same as that which was tested.

The current version of the ASTM Tentative Specification C 494 – 65 T recognizes each of the four needs. A test procedure is outlined using a blend of cement, aggregates of specified grading, and mixture proportions based on American Concrete Inst. (ACI) Recommended Practice for Selecting Proportions for Concrete (613-54), and it recommends that, where practicable, tests be made using the cement, aggregates, and air-entraining admixture proposed for the specific work. However, it contains no recommendations on an abridged set of tests which might serve to assure that a particular admixture previously subjected to all the specification tests and found satisfactory when tested with specific aggregates, air-entraining agent, and blend of cement would perform satisfactorily with the other concrete ingredients on a particular job. The materials covered by ASTM Specification C 494 are often found particularly useful at temperatures well below or above the laboratory temperature of 73 F required by the specification, and a few tests at temperatures anticipated in the field might be profitable.

The specification requires the manufacturer to recommend, upon request, appropriate test procedures for establishing the equivalence of materials from different lots. Qualitative, and to some extent quantitative, compositional analysis of water-reducing retarders may be obtained by infrared or ultraviolet absorption spectroscopy [*28*,*29*]. One alternate to this type of test consists of watching the rate of hardening, water requirement, and slump in the job concrete as use of a new lot of admixture is started, while another involves laboratory concrete tests using the new batch and a reserve sample from the initial batch [*30*].

The complete sets of tests prescribed in ASTM Specification C 494 is so extensive, and requires such an extended period of time, that it is unlikely that it will be performed more than a few times for any given admixture, and perhaps never for materials used in actual construction work. These tests are desirable and necessary to establish the potential value of an admixture, and the test data should be available to the purchaser, but as a practical matter, less extensive tests might well be prescribed for assessing the admixture's performance with the specific materials and conditions of given concrete work.

As presently written the specification cannot readily serve as a contractual document, because alternate procedures are given without the requirement that the alternate desired be specified. How would one judge, for example, if an admixture meeting all of the requirements of Specification C 494, Type A, when tested using laboratory materials and under laboratory conditions, meets the requirement of Paragraph 407 of the ACI Building Code (318-63) when its intended use is with a different cement and with, say, lightweight aggregates?

Since its introduction, the specification has been undergoing constant improvement and clarification, and these suggested minor deficiencies will no doubt be resolved.

In establishing specifications for concrete admixtures, allowance must be made not only for the statistical variation of test results on the concrete containing the admixture, but also that of the control concrete without admixture. For example, if it is intended that a water-reducing admixture increase the 28-day compressive strength by 20 per cent, and the limit therefore is set at 120 per cent of the control, an admixture which actually, on the average, will produce an increase of 20 per cent in strength would fail to meet the specification requirement 50 per cent of the time. The limits given in Table I of ASTM Specification C 494 take this factor into account by lowering the strength requirements and the water requirement by approximately 10 per cent. These figures were selected originally as a matter of good judgment, and they are subject to future change in the light of recent statistical considerations of the problem. The specification requirement for Type B (retarding) admixture that the strength of treated concrete at any age be at least 90 per cent of that of the untreated control has the objective of requiring that there be no sacrifice in strength due to use of the admixture. However, under the requirements of the specification, an admixture which actually caused a 10 per cent reduction in strength would be rejected, on the average, only 50 per cent of the time. The rate of rejection for true strengths above or below this point depends on the coefficient of variation of the test data and the number of test specimens. Protection to the consumer against acceptance of inferior products can be increased by improving test procedures so that the coefficient of variation is lowered, increasing the number of test specimens, or both.

One proposal, developed by Subcommittee II-a of ASTM Committee C-9, would retain the 90 per cent of control required where equal or better performance is intended, but would require 5 test specimens (rather than 3 as now specified for most tests) for both the control and the test concrete, and would require discarding the results if for either concrete the ratio of the range of the 5 results to the mean exceeds 0.35. Such a limitation on the test data would be satisfied 94 per cent of the time by data with a coefficient of variation of 10 per cent, but only 45 per cent of the time by data with a 15 per cent coefficient of variation. This limitation would, therefore, usually require data having a coefficient of variation not greater than 10 per cent.

With data of 10 per cent coefficient of variation, an admixture producing a true strength of only 90 per cent of the control would still, of course, be accepted 50 per cent of the time, but the probability of acceptance would drop rapidly for strengths below this level, being only 21 per cent for strengths of 85 per cent of the control, and 6 per cent for strengths of 80 per cent of the control. The probability of rejecting a material with true strength equal to the control would be 6 per cent.

The balance between probability of accepting an inferior product and rejecting a satisfactory one can be adjusted within limits, but substantial improvement can be achieved only by further reduction in the coefficient of variation through better testing and test methods, or the use of more test specimens. For further information on this matter reference should be made to the chapter on Evaluation of Test Results.

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Other Materials

ORGANIC MATERIAL FOR BONDING, PATCHING, AND SEALING OF CONCRETE

By M. R. Smith[1]

Concrete ranks very high among the commonly used construction materials. However, it is subject to attack by corrosive agents, including acids, salts, water, and organic solvents that wet exposed surfaces and penetrate into pores. Penetration of the concrete by water and aqueous solutions is especially important where the concrete is exposed to repeated cycles of freezing and thawing. In order to make the concrete more resistant to attack through these agencies, thereby increasing the life of the structure, it is sometimes coated with materials which protect the surface by preventing the ingress of water and other aggressive liquids into the concrete. Patches to replace defective or deteriorated areas are required frequently. Since concrete is weak in bond, the concrete to be patched is frequently coated with an adhesive that will promote bonding between the patching material and the hardened concrete.

Organic materials which have been used for bonding, patching, or sealing of concrete include epoxy resins, silicones, bitumens, linseed oil, oil-based paints, acrylics, urethanes, vinyls, rubber-base or resin-base coatings, and polyester resins. Epoxy resin systems are the most versatile for these uses.

Bonding and Patching Materials

Materials which, in recent years, have been used for bonding and patching of concrete fall into three general groups, namely: (1) admixtures, (2) bonding compounds (adhesives), and (3) resinous mortars.

Admixtures:

Admixtures used to improve bonding of new concrete to old are water emulsions of any of several organic materials, such as poly(vinyl acetate), acrylics, and butadiene styrene. These emulsions may be mixed with portland cement, mortar, or grout for application on the surface of the concrete just prior to placing of topping, patching, or surfacing material. The proper use of these materials is very important. The water emulsion systems are in many instances reemulsifiable; hence, such formulations should be avoided in areas subject to weathering or excessive moisture. Some emulsions are known to be stable under such conditions and should be considered for use where such conditions exist.

Surface Applications:

Epoxy resins are the most widely used of the organic systems for patching and bonding of concrete, both as an adhesive and when used as a binder for aggregate. Epoxy resins are polyethers usually

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[1] Bridgewater, Conn.

produced by reaction of epichlorohydrin (a relative of glycerine) and bisphenol A (a relative of carbolic acid), a derivative of natural gas or coal. A catalyst, usually an amine or polyamide (a group reaction of amine and fatty acids), is added to effect crosslinking of the resin molecules, thus producing setting and hardening. The set resin may have a tensile strength more than ten times that of concrete and a compressive strength more than double that of concrete. They are waterproof, resistant to many acids, bases, organic solvents, and mildew, and will remain stable at a wide range of temperatures [*1*].[2]

Repairs made where epoxy resin formulations are the bonding medium require only enough curing necessary to harden the epoxy and give it strength equal to that of the concrete. The bond strength, in this case, does not depend on the concrete curing.

At present there are no universally accepted specifications on epoxy resin systems for use with concrete. However, some states have developed and are using their own specifications. The California State Division of Highways was a pioneer in this field. At this writing, the American Society for Testing and Materials, through Committee C-9, is developing specifications which will be issued as a tentative standard in the near future. As now proposed, these specifications classify epoxy resin systems by type, class, grade, and color as follows:

1. for use in bonding hardened concrete and other materials to concrete,
2. for use in bonding freshly placed concrete to hardened concrete, and
3. for use in bonding skid-resistant materials to hardened concrete and as a binder in epoxy mortar or epoxy concrete.

Each type is divided into three grades based upon its flow characteristics, that is, Grade I has low viscosity, Grade II has moderate viscosity, and Grade III is for nonsagging application. These specifications define an epoxy resin as a resin that contains or did contain epoxy groups that are principally responsible for its polymerization. They also require that the epoxy resins shall be furnished in two components for combining immediately prior to use. Component A shall contain a bisphenol A, epichlorohydrin epoxy resin with or without a reactive diluent. Component B shall contain one or more curing agents which, on mixing with Component A, will cause the system to harden. A suitable inert filler may be uniformly incorporated in one or both components. The specifications will stipulate limits for viscosity, consistency, gel time, flexibility, creep, compressive strength, bond strength, and per cent of volatile matter.

The unmodified epoxy resin formulations are generally hard and brittle. For many applications more flexible, tougher formulations are needed. These can be obtained by adding an alloy or modifier. The principal modifiers are polysulfide polymers, polyamide polymers, and aliphatic amines. The polysulfide polymer modifier will produce a relatively resilient system with low shrinkage. The polyamide polymer cures the epoxy system because of the presence of amine groups in its structure. These react with the epoxide group to form tough, flexible compounds.

It should be pointed out that epoxy resin formulations either modified or unmodified can be obtained to fit most conditions. It is necessary to use the formulation recommended for a particular condition, that is, some formulations will not bond to a damp or wet surface, where others will. Until the ASTM specification covering epoxy systems to fit particular conditions is issued, the

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[2] The italic numbers in brackets refer to the list of references appended to this paper.

recommendations of the manufacturer should be requested to obtain formulations which will be satisfactory for those conditions.

The setting time, tack-free time, and curing time vary from a few minutes to several hours depending largely on the temperature and the curing compound used. Formulations can be varied to give different times as required.

For small patches, it may be advisable to use an epoxy resin mortar which is made by mixing sand or other inert filler with the epoxy resin to form an epoxy mortar which can be used to fill the hole or depression. Deeper areas may be filled with an epoxy resin concrete containing both fine and coarse aggregate. For patches on vertical surfaces it may be necessary to use a form to hold the mortar until it hardens. The instructions given in the ACI Guide [*2*], described hereinafter, apply to vertical as well as horizontal surfaces and to epoxy mortar as well as the unfilled epoxy compounds. Ordinary lubricants dissolve in and deteriorate surfaces of epoxy. Regular form oil should not be used to coat forms which come in contact with epoxy mortar. These form surfaces should be coated with special silicone lubricants, or form coverings of cellophane or polyethylene used. The amount of filler to be used will be that which will give a consistency adequate for filling the hole and preparing a satisfactory finish. Depending on the type of filler used, systems may contain up to 94 per cent of filler by weight. The amount of filler used is limited not only by the workability but also by the void content of the mortar or concrete. Excessive void content will prevent satisfactory finishing of exposed surfaces, and void content in excess of about 12 per cent by volume may seriously impair resistance of the hardened mixture to freezing and thawing.

Inert pigment fillers can be added to achieve color. Other inorganic powders are added to control viscosity, heat dissipation, and other properties. Fibrous-type fillers, sometimes called reinforcements, such as glass, asbestos, cotton, or synthetic fibers, are sometimes added [*3*].

American Concrete Institute Committee 403 [*2*] has issued a guide covering the use of epoxy resin in connection with concrete construction. This report should be studied by all planning to use these materials. It stresses the necessity for preparing the concrete surface to be treated before application of the resin system. Two surface conditions must be met if an application is to be successful: (1) the surface must be strong and sound, and (2) the surface must be dry and clean, that is, free from oil, grease, or other contaminants, such as residues of curing compounds, waxes, or polishes that may have been applied to the surface of the concrete. Modified epoxy resin systems may be applied under damp conditions.

The surface may be cleaned by any one of several methods. Probably the most common and most effective is sandblasting. Another method frequently used is acid etching, using muriatic acid. Oil or grease can be removed by scrubbing the surface vigorously with a strong detergent solution. All unsound concrete must be removed before application of the epoxy resin. Failure to properly clean the surface or to remove the unsound concrete will result in loss of bond. A method is described in the Report of ACI Committee 403 by which the effectiveness of the cleaning method can be evaluated.

In addition to preparation of the surfaces, other precautions must be observed in the use of epoxy resin systems. Thorough mixing of the components is essential, but more material than can be used within the pot-life of the material should not be prepared at any one time. The pot-life to be expected at various temperatures and with various proportions of the catalyst should be indicated in instructions provided by the manufacturer. If a larger batch is mixed than is recommended, the curing process is rapidly accelerated by the exothermic reaction associated with the setting process and very rapid hardening may occur. The correct amounts of the two components should be measured into the mixing container. At the time of application, the temperature of the epoxy compound should be within the limits recommended by the manufacturer, and the temperature of the surface and surrounding air should normally be at least 60 F. Most epoxy compounds bond satisfactorily only to dry surfaces, so that it is necessary that the surface be dry before application is made. Application equipment should either be disposable or arrangement should be made to clean tools and equipment in a suitable solvent wash, such as perchloroethylene trichloroethylene, high flash naphtha, or others, before the epoxy system has hardened. These solvents should be used for cleaning only and should normally not be added to the epoxy compound applied to the concrete.

Epoxy resins contain chemicals that are irritants to the skin, eyes, and respiratory system. In addition, some of the components may cause a form of dermatitis and will affect individuals to different degrees. In the event of direct contact with the clothing, the soiled clothing should be removed at once, and, if the clothing cannot be thoroughly cleaned, it should be disposed of. Epoxy compounds spilled on the body should be removed immediately with soap and water. If the epoxy comes into contact with the eyes, they must be flushed with large amounts of water, and a physician should be consulted at once. It is advisable to wear safety goggles when using epoxy resin systems. Solvents other than soap and water or water-soluble proprietary cleaners should not be used to remove the epoxy resin from the skin, since most solvents will only dilute the epoxy compound, aiding it in penetrating the skin. The solvents also dry out the skin and make future attack easier. Workers using the compound should be instructed in the proper use of the material; if this is done, there is little danger associated with their use. Fortunately, instances of sensitization are rare.

COATINGS AND IMPREGNATIONS

Bitumens:

Bitumens include both asphalt-base materials and coal tars. These must be applied in the thickness recommended by the manufacturer, which may vary from 0.005 to $\frac{1}{8}$ in. There is considerable difference between asphalts and coal tars, so that it is important that the proper material be selected for particular jobs. Bituminous coatings most commonly used are asphaltic materials dissolved in a solvent, with or without a filler. They are resistant to many corrosive materials but are attacked readily by solvents which dissolve the asphalt. Also, unless specially processed, asphaltic coatings are permeable to water. Coal tar coatings possess a high degree of water resistance; whenever water will be encountered, coal tar coatings should be considered. If exposed to direct sunlight or high temperatures, they have a tendency to crack and craze. The best use for coal tar is for protection of underground surfaces which may be exposed to water.

Linseed Oil:

Thirty-eight states are either regularly applying a flax-derived linseed oil antispalling compound to concrete surfaces, principally pavements, or are experimenting with its use [*4*]. Basically, there are two types of linseed oil antispalling compounds: (1) the solution type and (2) the emulsion type. The solution type is a mixture of polymerized linseed oil and mineral spirits (commonly in equal parts). The emulsion type contains polymerized linseed oil only, kerosene, detergent to keep the oil in suspension, trisodium phosphate, and water. The solution types are cheaper and more widely used. The protective effect obtained is due to penetration of the linseed oil into the concrete to depths of about $\frac{1}{8}$ in.; with hardening of the drying oil through combination with the oxygen of the air, an effective seal can be developed. The oxidized film prevents the entry of water and aggressive solutions. A metal dryer is frequently added to hasten the drying, which would otherwise take 40 to 50 hr. New concrete should be given a neutralizing wash to prevent saponification of the linseed oil. A solution of 0.25 lb of phosphoric acid to a gallon of water is brushed over the surface and allowed to dry for 48 hr before application of the linseed oil composition. This application is not necessary for old concrete [*5*].

Oil Paints:

The oil paint coatings are normally a combination of linseed oil, a pigment, and a dryer [*6,7*]. The drying oil is a reactive material that absorbs oxygen from the air and reacts with it in order to dry. These coatings are inexpensive and are easily applied. They are flexible and durable when exposed to the weather. They have low chemical and corrosive resistance, and their use is restricted to those surfaces which are not exposed to corrosive agents. On new concrete, a neutralizing wash should be applied to avoid saponification of the oil.

Acrylics:

Acrylics are esters of acrylic or methacrylic acid. These materials, as with bitumens and linseed oil, are applied to dry concrete and are generally designed to penetrate and form a moisture barrier in the pores. They may be formulated to a relatively heavy consistency in which condition they do not penetrate but form a surface film which is an effective moisture barrier. The acrylics used as penetrants have only a slight effect on color of the concrete with aging. When used as a surface coating, the color upon aging may be amber. Acrylic latexes have proven to be the most decorative coating for concrete. Acrylic latexes differ from the solvent solution acrylic lacquers in that the latexes allow passage of vapor.

Rubber-Base or Resin-Base Coatings:

Numerous resins have been developed for application as solutions in various solvents to provide excellent chemical resistance of concrete surfaces. Among these resins are coumarone-indene, styrene-butadiene, chlorinated rubber, chlorosulfonated polyethylene, and neoprene. These materials are relatively inexpensive and easy to apply. They can be brushed or sprayed onto the surfaces and are resistant to water. Their resistance to alkalies makes them excellent for use on concrete. Ultraviolet radiation in sunlight accelerates dechlorination of chlorinated rubber, resulting in color changes and breakdown of structure [*8*]. This effect can be reduced by use of suitable pigments and chemicals known as ultraviolet light absorbers.

Most rubber-base coatings are actually resin-base coatings. The rubber is in many cases one which has been modified to form a resin. For example, by adding more styrene to a styrene-butadiene rubber latex, a hard, brittle, tough resin is obtained. Similarly, when natural rubber is heavily chlorinated, the resultant product is chlorinated rubber, which is actually a resin.

Urethanes:

Urethanes are reaction products of an isocyanate and an alcoholic hydroxyl group. These coatings are not as resistant as epoxy resins to many chemicals. They are satisfactory for many uses, including those where exterior durability and abrasion resistance are important. Their color retention is low, and these materials may not be suitable if decoration as well as protection is important. They are supplied as two-part systems, the resin and a catalyst, for intermixture just prior to application, or as one-part systems, the polymerization occurring as a result of interaction with moisture from the air or the substrate. The reaction with water is accompanied by release of carbon dioxide gas. Urethane coatings require careful surface preparation and meticulous attention to the manufacturer's recommendations.

Polyesters:

Polyesters are reaction products of a difunctional alcohol or an anhydride of dibasic acid. The polyester coatings are noted for their color and color retention. Their chemical resistance is not as good as that of epoxy resins or urethanes. Polyester coatings also require careful surface preparation, and the manufacturer's recommendations must be followed carefully if satisfactory results are to be obtained. Many polyesters cannot be bonded directly to portland cement concrete.

Silicones:

Silicones are high-molecular-weight organosilicon compounds. As used for coatings for concrete, they are dilute water solutions of such water-soluble compounds as sodium methyl siliconate, water-based emulsions of silicone resins, or mineral-solvent solutions, usually at concentrations in the range 2 to 5 per cent. These coatings differ from other coatings in that they do not form a film. They are applied to produce water repellency of surfaces and to decrease the rate of absorption of water and aggressive solutions. Effectiveness of such coatings in improving resistance of exposed concrete to effects of freezing and thawing is controversial [*9*,*10*].

Silicones that must be heated to accomplish setting are used where resistance to high temperature is important, such as in chimneys. Setting of the coating is accomplished by heating of the stack.

Vinyls:

Vinyls are copolymers of vinyl chloride and acetate. Sarans and vinylidene chloride are also classified as vinyls. Both of these materials are thermoplastic, and their resistance to most corrosive agents is very high. In solution form they have low solid content so that multiple coatings are required to obtain adequate film thickness. When proper film thickness is obtained, their resistance is excellent, except to certain organic solvents. Many vinyls also suffer poor adhesion, and it may be necessary to apply a specially prepared primer to assure adequate bond to the surface of the concrete.

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