Designation: C457/C457M – 16

Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete[1]

This standard is issued under the fixed designation C457/C457M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method describes procedures for microscopical determinations of the air content of hardened concrete and of the specific surface, void frequency, spacing factor, and paste-air ratio of the air-void system in hardened concrete. Three procedures are described:

1.1.1 *Procedure A*—Linear-traverse method.

1.1.2 *Procedure B*—Modified point-count method.

1.1.3 *Procedure C*—Contrast enhanced method.

1.2 This test method is based on prescribed procedures that are applied to sawed and lapped sections of specimens of concrete from the field or laboratory.

1.3 It is intended to outline the principles of this test method and to establish standards for its adequate performance but not to describe in detail all the possible variations that might be used to accomplish the objectives of this test method.

1.4 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.5 *This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements see Note 9 *and* Note 12.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C42/C42M Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete

C125 Terminology Relating to Concrete and Concrete Aggregates

C138/C138M Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete

C173/C173M Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method

C231/C231M Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method

C666/C666M Test Method for Resistance of Concrete to Rapid Freezing and Thawing

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

C672/C672M Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals

C823/C823M Practice for Examination and Sampling of Hardened Concrete in Constructions

C856 Practice for Petrographic Examination of Hardened Concrete

D92 Test Method for Flash and Fire Points by Cleveland Open Cup Tester

2.2 *American Concrete Institute Standards:*[3]

201.2R Guide to Durable Concrete

211.1 Standard Practice for Selecting Proportions for Normal, Heavyweight, and Mass Concrete

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology C125.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *average chord length* $(\bar{l})$, *n*—the average length of the chords formed by the transection of the voids by the line of

[1] This test method is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.65 on Petrography.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American Concrete Institute (ACI), P.O. Box 9094, Farmington Hills, MI 48333-9094, http://www.aci-int.org.

*A Summary of Changes section appears at the end of this standard traverse; the unit is a length.

3.2.2 *binary image, n*—formed by segmenting an image using only one threshold with the resulting image having only areas of black or white.

3.2.3 *digital image, n*—an image captured using a computer-based storage method where the information presented in the image can be seen visually, like a traditional photographic image, but can also be extracted in a numeric form that can be used for additional analysis.

3.2.4 *paste-air ratio (p/A), n*—the ratio of the volume of hardened cement paste to the volume of the air voids in the concrete.

3.2.5 *paste content (p), n*—the proportion of the total volume of the concrete that is hardened cement paste expressed as percentage by volume.

3.2.5.1 *Discussion*—When this parameter is calculated, it is the sum of the proportional volumes of the cement, the net mixing water (including the liquid portions of any chemical admixtures), and any supplementary cementitious materials present.

3.2.6 *pixel, n*—the smallest definable point of a digital image that has an assigned value representing the brightness of that component in an image.

3.2.6.1 *Discussion*—Typically a pixel will have the same aspect ratio as the overall image and will have an assigned integer value occurring in the range 0-255. Multiple pixels are arranged contiguously in two-dimensional arrays to form a digital image.

3.2.7 *segment, v*—the process of placing image pixels into classes or like-groupings using any number of thresholds.

3.2.8 *spacing factor* $(\bar{L})$, *n*—a parameter related to the maximum distance in the cement paste from the periphery of an air void, the unit is a length.

3.2.9 *specific surface (α), n*—the surface area of the air voids divided by their volume, expressed in compatible units so that the unit of specific surface is a reciprocal length.

3.2.10 *threshold, n*—a value used to discriminate pixels into more than one class or like grouping.

3.2.11 *void frequency (n), n*—voids per unit length of traverse; the number of air voids intercepted by a traverse line divided by the length of that line; the unit is a reciprocal length.

3.2.11.1 *Discussion*—The value for void frequency *(n)* cannot be directly determined by the paste-air ratio method as this value refers to the voids per unit measure of traverse in the total concrete (including aggregate).

3.2.12 *water void, n*—a space enclosed by the cement paste that was occupied by water at the time of setting and frequently found under an aggregate particle or reinforcing bar. A water-void is usually identified by its irregular shape or evidence that a channel or cavity has been created by bleed water trapped in the concrete at the time it hardened.

4. Summary of Test Method

4.1 *Procedure A, Linear-Traverse Method*—This procedure consists of the determination of the volumetric composition of the concrete by summing the distances traversed across a given component along a series of regularly spaced lines in one or more planes intersecting the specimen. The data gathered are the total length traversed (T_t), the length traversed through air voids (T_a), the length traversed through paste (T_p), and the number of air voids intersected by the traverse line *(N)*. These data are used to calculate the air content and various parameters of the air-void system. If only the air content is desired, only T_a and T_t need be determined.

4.2 *Procedure B, Modified Point-Count Method*—This procedure consists of the determination of the volumetric composition of the concrete by observation of the frequency with which areas of a given component coincide with a regular grid system of points at which stops are made to enable the determinations of composition. These points may be in one or more planes intersecting the specimen. The data gathered are the linear distance between stops along the traverse *(I)*, the total number of stops (S_t), the number of stops in air voids (S_a), the number of stops in paste (S_p), and the number of air voids *(N)* intersected by the line of traverse over which the component data is gathered. From these data the air content and various parameters of the air-void system are calculated. If only the air content is desired, only S_a and S_t need be determined.

4.3 *Procedure C, Contrast Enhanced Method*—This procedure consists of the determination of the volumetric composition of the concrete by summing distances measured in digital images of a prepared concrete surface using a series of regularly spaced lines in one or more digital images obtained from one or more planes intersecting the specimen. The specimen is prepared exactly as described for Procedures A and B with the additional steps of darkening the specimen surface and filling the air voids with a fine particle size white powder. The data gathered are the total length measured (T_t), the length measured through air voids (T_a), and the number of air voids intersected by the measurement lines *(N)*. These data are used to calculate the air content and various parameters of the air-void system, except the paste-air ratio and spacing factor that require determination of the paste content (T_p) as described in 16.1. If only the air content is desired, only T_a and T_t need be determined.

4.4 *Paste-Air Ratio Modification*—In some instances the specimen is not representative of the concrete as a whole, so T_t and S_t lose their significance and cannot be used as a basis for calculations. The most common examples are concrete with large coarse aggregate and specimens from the finished surface region, for both of which the examined specimen consists of a disproportionately large amount of the mortar fraction. In such instances the usual procedure must be changed, and the paste-air ratio modification must be used (see 5.5).

5. Significance and Use

5.1 The parameters of the air-void system of hardened concrete determined by the procedures described in this test method are related to the susceptibility of the cement paste portion of the concrete to damage by freezing and thawing. Hence, this test method can be used to develop data to estimate the likelihood of damage due to cyclic freezing and thawing or to explain why it has occurred. The test method can also be used as an adjunct to the development of products or procedures intended to enhance the resistance of concrete to cyclic freezing and thawing.

5.2 Values for parameters of the air-void system can be obtained by any of the procedures described in this test method. The selection of which one of the three methods to be used shall be subject to agreement of the user and provider of the determination

NOTE 1—Because Procedure C requires darkening the paste and aggregate, its use must occur after other tests if the analyst is also gathering petrographic data in addition to the measurements described in this test method.

5.3 No provision is made for distinguishing among entrapped air voids, entrained air voids, and water voids. Any such distinction is arbitrary, because the various types of voids intergrade in size, shape, and other characteristics. Reports that do make such a distinction typically define entrapped air voids as being larger than 1 mm in at least one dimension being irregular in shape, or both. The honey-combing that is a consequence of the failure to compact the concrete properly is one type of entrapped air void.

5.4 Water voids are cavities that were filled with water at the time of setting of the concrete. They are significant only in mixtures that contained excessive mixing water or in which pronounced bleeding and settlement occurred. They are most common beneath horizontal reinforcing bars, pieces of coarse aggregate and as channelways along their sides. They occur also immediately below surfaces that were compacted by finishing operations before the completion of bleeding.

5.5 Application of the paste-air ratio procedure is necessary when the concrete includes large nominal maximum size aggregate, such as 50 mm [2 in.] or more. Prepared sections of such concrete should include a maximum of the mortar fraction, so as to increase the number of counts on air voids or traverse across them. The ratio of the volume of aggregate to the volume of paste in the original mix must be accurately known or estimated to permit the calculation of the air-void systems parameters from the microscopically determined paste-air ratio.

NOTE 2—The air-void content determined in accordance with this test method usually agrees closely with the value determined on the fresh concrete in accordance with Test Methods C138/C138M, C173/C173M, or C231/C231M. However, significant differences may be observed if the sample of fresh concrete is consolidated to a different degree than the specimen later examined microscopically. For concrete with a relatively high air content (usually over 7.5 %), the value determined microscopically may be higher by one or more percentage points than that determined by Test Method C231/C231M.

SAMPLING AND SECTION PREPARATION

6. Apparatus and Materials for Specimen Preparation

6.1 *Apparatus and Materials for All Procedures—*

6.1.1 Apparatus and materials for the preparation of surfaces of concrete speciemens for microscopical observation are described in Practice C856; other apparatus may be equally suitable.

6.2 *Materials for Procedure C—*

6.2.1 *Opaque Permanent Black Ink,* wide felt-tipped marker, black ink stamp pad, or black ink roller, or similar.

6.2.2 *White Powder,* barium sulfate, wollastonite, or titanium dioxide with a median particle size of 2-3 µm, or similar.

6.2.3 *Light Oil,* light mineral oil, or similar.

NOTE 3—Apparatus for measurement of prepared specimens is described in the three following procedures.

7. Sampling (for all procedures)

7.1 Specimens of concrete can be obtained from concrete cast in the field or laboratory, or by coring, sawing, or otherwise removing concrete from structures or products. The procedure followed and the location from which the specimens are obtained will depend on the objectives of the program. In general, secure samples of hardened concrete in accordance with Test Method C42/C42M or Practice C823/C823M or both. Provide at least the minimum area of finished surface given in Table 1 in each specimen. A sample may be composed of any number of specimens.

7.2 For referee purposes or to determine the compliance of hardened concrete with requirements of specifications for the air-void system, obtain samples for analysis by this test method from at least three randomly selected locations over the area or throughout the body of concrete to be tested, depending upon the objectives of the investigation.

8. Preparation of Sections

8.1 *Preparation of Sections for All Procedures—*

8.1.1 Unless the objectives of the program dictate otherwise, saw the section for observation approximately perpendicular to the layers in which the concrete was placed or perpendicular to the finished surface. Individual sections

TABLE 1 Minimum Area of Finished Surface for Microscopical Measurement[A],[B]

Nominal or Observed Maximum Size of Aggregate in the Concrete, mm [in.]	Total Area to be Traversed[C] for Determination of p,[D]A, α, or $\bar{L}$, min, cm^2 [in.^{2}] Based on Direct Measurement of:	
	Total Air-Void Content	Paste-Air Ratio, p/A
150 [6]	1613 [250]	645 [100]
75 [3]	419 [65]	194 [30]
37.5 [1½]	155 [24]	97 [15]
25.0 [1]	77 [12]	77 [12]
19.0 [¾]	71 [11]	71 [11]
12.5 [½]	65 [10]	65 [10]
9.5 [⅜]	58 [9]	58 [9]
4.75 (No. 4)	45 [7]	45 [7]

[A]The indicated values refer to reasonably homogeneous, well-compacted concrete. The microscopical measurement shall be made on proportionately larger area of sections if the concrete is markedly heterogeneous in distribution of aggregate or large air voids. If more than one finished surface is taken from a single portion of the concrete, the finished surfaces shall be separated by a distance greater than one half of the nominal or observed maximum size of aggregate.

[B] See Section 3 for the interpretation of symbols employed.

[C]When performing a point count to determine p, A, α, or $\bar{L}$, the analysis points shall be distributed evenly over the area to be traversed.

[D]When p is determined, it shall be determined by analyzing the same area to be traversed for determination of A, α, or $\bar{L}$.

should be as large as can be ground and examined with the available equipment. The required area may consist of more than one prepared section. Spread the selected traverse length uniformly over the available surface so as to compensate for the heterogeneity of the concrete.

8.1.2 If gross irregularities are present, begin the surface preparation by lapping (grinding on a flat surface) with nominal 150 µm (No. 100) silicon carbide abrasive. Lap the surface with successively finer abrasives until it is suitable for microscopical observation. An appropriate series of abrasives would include nominal 75, 35, 17.5 and 12.5 µm grit sizes (No. 220, 320, 600, and 800, respectively), and perhaps 5-µm (No. 2500 grit) aluminum oxide.

NOTE 4—Grit numbers of abrasives can denote slightly different particle sizes, depending on the manufacturer. The suggested sizes will usually be appropriate, but others may be selected according to the experience of the user.

From time to time during lapping, and when changing to a finer abrasive and when lapping is complete, clean all surfaces of the specimen gently and thoroughly to remove the grinding compound. Use of ultrasonic cleaners may be harmful to the surface. Such treatment should not be used without care and experimentation. Cleaning with a soft cosmetic brush under running water, or by a pressurized dental spray has been successful. A surface that is satisfactory for microscopical examination will show an excellent reflection of a distant light source when viewed at a low incident angle and there shall be no noticeable relief between the paste and the aggregate surfaces. Areas that are scratched or imperfect indicate the need for additional preparation; use special techniques if required (see Note 9 and Note 12). The edges of the sections of the air voids will be sharp and not eroded or crumbled, and air-void sections including those as small as 10 µm [0.0004 in.] in diameter will be clearly distinguishable. (See Fig. 1.) Do not include scratched or broken portions of the surface in the analyzed area. If needed to meet the requirements of Table 1, prepare additional surfaces.

8.1.3 Sometimes difficulty will be encountered in preparing the lapped surfaces. The usual cause is a weak cement-paste matrix. The problem is manifested by the plucking of sand grains from the surface during the lapping, with consequent scratching of the surface, and by undercutting of the paste around the harder aggregate particles. Friable particles of aggregate can also cause difficulty. In such instances the

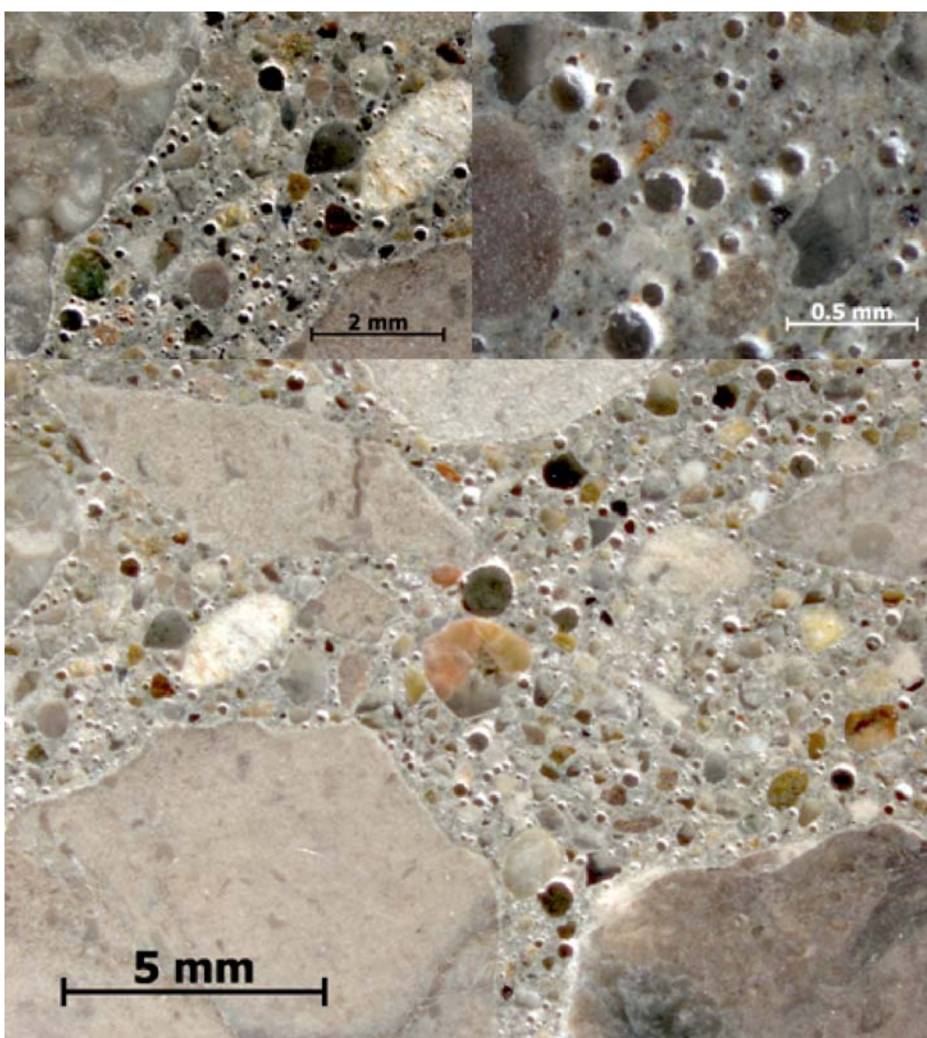


FIG. 1 Photographs of a Satisfactory Surface

following procedure is helpful. Heat the partially prepared specimen of concrete to about 150 °C [300 °F] in an oven. (**Warning—**If the specimen was sawn with a lubricant other than water, heating must be done so as to avoid inhaling the fumes and to preclude fire or explosion. Some lubricants have a flash point as low as 140 °C [285 °F]. (The flash point of the lubricant may be found by use of Test Method D92.) Unless other precautions are taken, the temperature must not be allowed to approach the flash point. If this cannot be avoided, heating must be done in the open air on a hot plate or in an explosion-proof hood.)

Remove the specimen from the oven and immediately brush melted carnauba wax that was heated to the same temperature onto the surface. Repeat the application as the wax is absorbed by the concrete, so that when the temperature of the concrete falls below the melting point of the wax, a perceptible film remains on the surface. After the specimen has cooled, scrape off any excess wax and repeat the lapping. After completion of lapping, remove the residue of wax from the surface air voids by reheating the concrete to about 150 °C [300 °F] to allow absorption of the molten wax into the specimen. Again take care to avoid approaching the flash point of the wax or of any cutting oil present. Protect the surface from dust during heating. The time to remove the wax from the surface air voids varies with the properties and thickness of the specimen, but heating for about an hour is usually sufficient. Exceptionally fragile concrete may require repetition of this process. Substances other than carnauba wax have been used successfully to impregnate and strengthen the surfaces of concrete specimens before grinding.

NOTE 5—If performing a microscopic examination of the cement paste, using Practice C856, on the same test specimen that will be coated with wax or other paste strengthening media, perform the microscopic examination prior to heating the specimen and application of the strengthening materials. The application of such materials and exposure to oven temperatures of 150 °C [300 °F] will alter the physical characteristics and appearance of the cement paste.

8.1.4 If the parameters of the air-void system near a finished or formed surface are desired, then prepare the section examined in such a manner as to allow for the fact that the parameters of the air-void system may vary greatly with the distance from such a surface. Therefore, measure the distance between the section to be examined and the original surface accurately, to at least the nearest 1 mm [0.05 in.]. Use the following procedure: (*1*) Prepare a specimen that includes a portion of the finished or formed surface to be investigated, and of convenient thickness, but not less than 12 mm [½ in.] or one-half of the nominal maximum size of the aggregate, whichever is greater. (*2*) Lap the surface with a coarse abrasive until the last portion of the original surface is just removed, then complete the lapping operation as described above. Use this surface as the reference plane, to which later measurements are referenced. (*3*) Lap the back surface of the specimen so as to produce a plane section. (*4*) Measure the thickness of the specimen to the nearest 1 mm [0.05 in.] at four or more points uniformly spaced around the periphery. Average the results, and record the average to the nearest 1 mm [0.05 in]. (*5*) Determine the parameters of the air-void system on any plane desired or specified. If nearest surface values are desired, make the determination on the reference plane; if values for the bulk concrete are desired, make the determination on the back plane. If values for some other plane are desired, repeat the grinding process to the desired depth. Redetermine the thickness of the specimen as specified above so that the parameters of the air-void system can be correlated with the distance of the examined surface from the reference plane.

8.1.5 The composition of the near-surface zone differs from that of the concrete as a whole. Therefore, whenever the design of the mixture is known, use the paste-air ratio method for the determination of the air-void system parameters in this region.

8.2 *Additional Preparation Required for Procedure C—*

8.2.1 Prepare specimens in accordance with 8.1.

NOTE 6—If paste-air ratio and spacing factor are to be determined, it is necessary to provide a determination of the specimen paste content. If this determination is to be made by point counting or another procedure requiring visually distinguishing aggregate from paste, such determinations must be performed prior to blackening the specimen surface.

8.2.2 Apply an even layer of opaque, permanent, black ink to the surface of the prepared specimen so the entire surface is rendered uniformly black.

NOTE 7—Black markers have successfully been used as has black ink applied with a roller such as that used in offset printing.

8.2.3 After the ink has dried, distribute a layer of white powder over the blackened surface and gently press it into the voids using a suitable implement such as a rubber stopper, glass rod, or petrographic slide. Avoid abrading the surface by minimizing rubbing the powder over the surface.

8.2.4 Remove excess powder by scraping it from the surface with a suitable implement (e.g., a sharp, new single-sided razor blade). Avoid abrading the ink thereby exposing cement or aggregate grains.

8.2.5 Inspect the darkened and filled surface. Areas where ink has been abraded should be re-touched with a local, fresh application of ink. Porous aggregate grains should likewise be re-touched with a local, fresh application of ink such that internal pores in the aggregate are rendered black. Blacken areas filled in by white powder that are identified as pullouts occurring during specimen preparation. Likewise, blacken areas filled in with white powder that are identified as coarse cracks or other defects in the specimen not classified as part of the air-void system.

8.2.6 With a very lightly oiled finger or thumb, remove the last remnants of white powder from the surface, leaving a shiny black surface without the presence of unfilled voids.

PROCEDURE A—LINEAR TRAVERSE METHOD

9. Apparatus for Measurement of Specimens

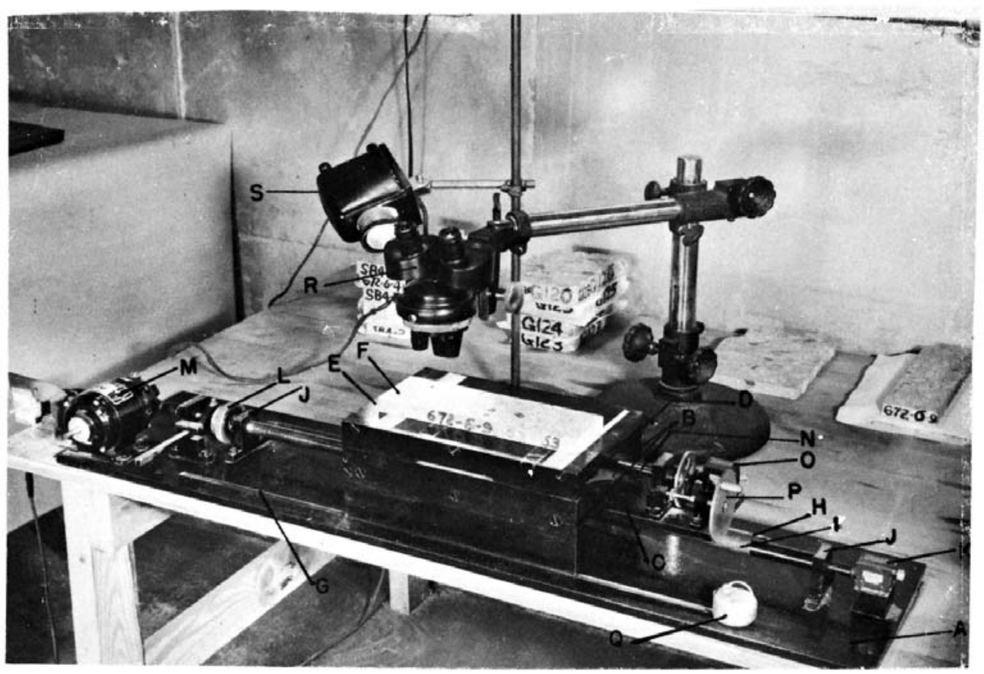
9.1 The apparatus listed in 9.1.1 to 9.1.5 comprises a recommended minimum selection. Apparatus other than that described has been used and may be equally satisfactory. Apparatus that uses electronic switches and totalizers has been constructed. Computerized apparatus is commercially available. Image analyzers have frequently been used.

9.1.1 *Linear-Traverse Device*—Provide a platform, on which the specimen is carried mounted on lead screws by means of which it can be smoothly translated in two perpendicular directions. Provide one lead screw for movement in the N-S direction and at least two for movement in the E-W direction.

NOTE 8—In the descriptions of the linear-traverse and point-count devices the term "E-W direction" refers to the direction from the operator's right to his left, and "N-S" means the direction perpendicular to E-W, that is, the directions are analogous to those on a map.

One of these latter is called the "main" lead screw and the other(s) the "upper" lead screw(s). Ensure that the capacity of the main (E-W) lead screw is at least 100 mm [4 in.], that of each (E-W) lead screw at least 65 mm [2.5 in.], and that of the N-S lead screw at least 75 mm [3.0 in.]. Ensure that the pitch of the upper lead screw does not exceed 0.265 mm [0.0105 in.] per revolution. Determine the pitch of all E-W lead screws to the nearest 0.025 mm [0.001 in.]. Attach rotation counters readable to the nearest 0.01 of revolution to all E-W lead screws. Provide a manually operated tally counter. For the determination of the paste content, provide a third E-W lead screw complete with rotation counter, unless each traverse is to be repeated, that is, performed once for the air content and again for the paste content. Photographs of satisfactory linear-traverse devices are shown in 3.

9.1.2 *Stereoscopic Microscope and Support,* with objectives and eyepieces to give final magnification in the range from about 50× to about 125×. While it is possible to use a



A = Base plate.
B = Front and back rails supporting the middle plate *C*
C = Middle plate.
D = Upper front and back rails carrying the stage *E*
E = Stage.
F = Concrete specimen
G = Rectangular front groove in the base plate.
H = V-shaped back groove in the base plate.
I = Main lead screw.
J = Two bearing blocks for the main lead screw.
K = Revolution counter on main lead screw.
L = Manually operated knurled wheel.
M = Electric motor for driving the main lead screw.
N = Upper lead screw.
O = Revolution counter for upper lead screw.
P = Hand-driven wheel for moving the stage.
Q = Ratchet counter to tally the number of air voids encountered.
R = Stereoscopic microscope.
S = Microscope lamp.

NOTE 1—Not shown are a third lead screw and a disengaging clutch; the former is necessary if a determination of the air-paste ratio is required, and the latter may be required (see 9.1.1).

FIG. 2 Photograph of a Linear-Traverse Device Meeting the Requirements of This Test Method

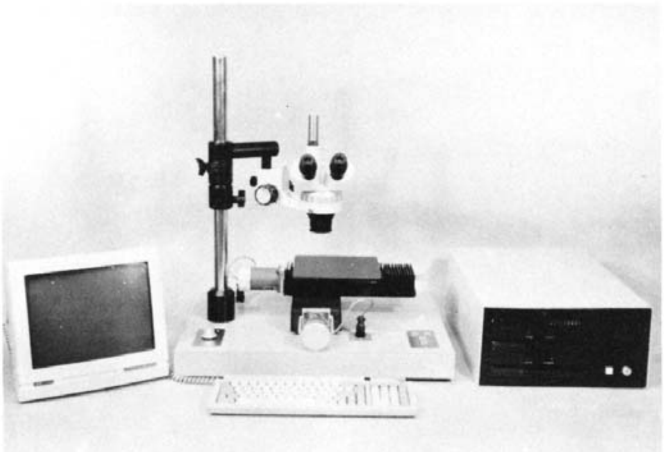


FIG. 3 Photograph of a Computerized Linear-Traverse/Point-Count Device Meeting the Requirements of This Test Method

microscope with a single, fixed magnification, it is more convenient to be able to vary the magnification within the above range by replacing eyepieces or objectives or, better, by means of a zoom attachment. Arrange the microscope so as to permit continuous observations of the surface of the specimen mounted on the platform of the linear-traverse device. Include cross hairs, scale, or some other reticle device to provide an index point in one eyepiece. Since an index point is dimensionless it shall be a point such as the intersection of one pair of edges of the cross hairs or one corner of the end of a line of a scale. Use the same index point throughout any examination.

9.1.3 *Microscope Lamp,* spotlight-type, arranged to provide sufficient illumination at a low and variable incident angle to the surface. The spot of evenly lit area on the specimen surface should be slightly larger than the field of view of the microscope.

9.1.4 *Spirit Level,* the small circular type is convenient.

9.1.5 *Leveling Device*—Provide a means to level the examined surface. This can be done by the insertion of small pieces of modeling clay. A better way is by means of a platform that is mounted on three adjustable leveling screws and that supports the specimen on the stage of the traverse device.

10. Procedure

10.1 Place the prepared specimen of concrete on the stage of the linear-traverse device. Level the prepared surface with the leveling device and the spirit level so that the surface may be traversed and microscopically examined with a minimum of refocusing. Adjust the lamp so that the beam evenly illuminates the field of view of the microscope and is incident on the surface at a low angle, so the air voids are demarked by a shadow. Superimpose the index point on the surface to be examined. Do not use a magnification of less than 50× and do not change it during the course of the analysis. For a rectangular section, place the index near an upper corner; for a circular section, place it near the top and at one end of the initial traverse. Engage and adjust all drives so as to remove all play from the gear systems. Set all counters to zero. By operation of the main lead screw move the assembly and specimen in the E-W direction while scrutinizing the prepared surface as it moves beneath the microscope.

NOTE 9—Surfaces examined may exhibit features that resemble air voids, but are not: *(a)* Occasionally a transparent section of a grain of quartz sand will look like an air void. *(b)* The socket left when a section of a nearly spherical and smooth sand grain is lost from the surface during grinding looks much like an air void, but can be distinguished by differences in the luster and sheen of the film lining the hole. *(c)* Cenospheres, hollow particles of fly ash, or hollow plastic spheres will also have a different sheen, and are unaffected when the surrounding paste is etched with dilute (10 %) hydrochloric acid.

Warning—Do not acid-etch the specimen under the microscope, as the effervescent spatter may damage the lens.

NOTE 10—Occasionally, air voids may become filled, during the service exposure of the concrete, with secondary products. Whether such voids are counted as belonging to the air-void system or not depends on the purposes of the investigation.

When the index point is exactly superimposed on the periphery of a section of an air void in the prepared surface of the specimen, stop the movement of the carriage, actuate the tally counter once, and by means of the upper lead screw, move the concrete under the microscope until the index point is exactly superimposed upon the opposite periphery of the same air-void section. Stop the rotation of the upper lead screw, and resume the movement by means of the main lead screw. Take extreme care to determine whether or not a section of an air void is intersected by the index point when the line of traverse is nearly tangent to the void section. The results can be affected significantly by consistent error in this respect. If the periphery of an air void is crumbled or rounded, estimate the position of the true periphery in the plane of the surface by extrapolation of the surface contour of the air void. If the paste content is being determined, as will usually be the case, carry out the above procedure for traverses across paste regions, except use the second upper lead screw and do not use the tally counter. Proceed in this way along the E-W traverse line, traversing all chords across air voids with the upper lead screw, all sections of paste with the second upper lead screw (if paste content is being determined), and all other sections with the main lead screw. Stop the traverse at the end of the line, which should be just within the examined area, not at its edge. By means of the N-S lead screw shift the specimen an appropriate distance to the next traverse line. Space the segments of the traverse so as to cover the entire prepared surface with at least the minimum required traverse length. If the rotation counters operate in both directions, the next line of traverse can begin just below the end of the previous one; if not, return the stage so the new line will begin just below the beginning of the previous one. Start each segment of the traverse just within the prepared area and on the satisfactory plane surface of the specimen rather than at the edge of the surface itself. The length of the segments of the traverse may vary. Superimpose the index point at the beginning of the new line, and perform the traverse as before. Repeat this process for all segments of the total traverse. Accumulate the total rotation on each counter, or read and record each at the end of each traverse line, so that the total will be the summation of such records. If more than one specimen has been prepared from the specimen of concrete, repeat the procedure on each such specimen as to comply with the requirements of Table 1. Electronic or computerized equipment will require that the procedures specified by the fabricator be followed but the principles will remain as detailed above. The minimum length of traverse shall be as specified in Table 2.

11. Calculation

11.1 When based on the air content of the total concrete:

11.1.1 The data will consist of:

where:

N = total number of air voids intersected,
R_i = number of rotations of the respective lead screws, and
P_i = pitch of the corresponding lead screws.

11.1.2 Calculate:

$$T_t = \text{total length of traverse} = \text{sum of } P_i \times R_i \tag{1}$$

$$T_a = \text{traverse length through air} = P_a \times R_a \tag{2}$$

$$T_p = \text{traverse length through paste} = P_p \times R_p \tag{3}$$

11.1.3 *Air Content (A),*in %:

$$A = \frac{T_a \cdot 100}{T_t} \tag{4}$$

11.1.4 *Void Frequency (n):*

$$n = \frac{N}{T_t} \tag{5}$$

11.1.5 *Average Chord Length* $(\bar{l})$:

$$\bar{l} = \frac{T_a}{N} \tag{6}$$

or

$$\bar{l} = \frac{A}{100n} \tag{7}$$

11.1.6 *Specific Surface* (α):

$$\alpha = \frac{4}{\bar{l}} \tag{8}$$

or

$$\alpha = \frac{4N}{T_a} \tag{9}$$

11.1.7 *Paste Content (p),*in %:

$$p = \frac{T_p \cdot 100}{T_t} \tag{10}$$

11.1.8 *Paste-Air Ratio (p/A):*

$$\frac{p}{A} = \frac{T_p}{T_a} \tag{11}$$

11.1.9 *Spacing Factor* $(\bar{L})$:

11.1.9.1 When *p/A* is less than or equal to

$$4.342\ \bar{L} = \frac{T_p}{4N} \tag{12}$$

11.1.9.2 When *p/A* is greater than

$$4.342\ \bar{L} = \frac{3}{\alpha}\left[1.4\left(1+\frac{p}{A}\right)^{1/3} - 1\right] \tag{13}$$

11.2 If the calculations are based on the paste-air ratio method, the design of the mixture must be known. The microscopically determined data will consist of T_p, T_a, and N. Proceed as follows:

11.2.1 Calculate the *paste-air ratio (r):*

$$r = \frac{T_p}{T_a} \tag{14}$$

11.2.2 From the mixture design calculate the ratio of *aggregate volume* to *paste volume (M):*

$$M = \frac{G_c}{p_c} \tag{15}$$

where:

G_c = sum of the masses of the aggregates; each divided by its specific gravity, and
p_c = sum of mass of cement divided by specific gravity of cement + mass of mineral admixture divided by the specific gravity of the mineral admixture + mass of water (including the fluid portion of any admixture) divided by the specific gravity of the fluids:

NOTE 11—The values used for the calculated G_c and p_c must accurately reflect the differing densities of the constituents and the proportions of the masses of each.

11.2.3 The *percent air (A):*

$$A = \frac{100}{r(1+M)+1} \tag{16}$$

11.2.4 *Paste* expressed as percent *(p):*

$$p = A \cdot r \tag{17}$$

11.2.5 *Average chord length* $(\bar{l})$:

$$\bar{l} = \frac{T_a}{N} \tag{18}$$

11.2.6 *Specific Surface* (α): Use Eq 8 and 9.

TABLE 2 Minimum Length of Traverse for the Linear Traverse Method[A]

Nominal or Observed Maximum Size of Aggregate in the Concrete, mm [in.]	Length of Traverse for Determination of A, α, or $\bar{L}$, min, mm [in.]
150 [6]	4064 [160]
75 [3]	3048 [120]
37.5 [1½]	2540 [100]
25.0 [1]	2413 [95]
19.0 [¾]	2286 [90]
12.5 [½]	2032 [80]
9.5 [⅜]	1905 [75]
4.75 (No. 4)	1397 [55]

[A] The limits of uncertainty of results obtained for air-void content depend upon the length of traverse and the air-void content of the concrete. Based on experience, the recommended minimum length of traverse shown in this table should produce limits of uncertainty such that up to 3 % air-void content the standard deviation is not greater than 0.5 %, which at 3 % air-void content corresponds to a coefficient of variation of 17 %. For traverse lengths greater than 1375 mm [55 in.] and air-void contents greater than 3 % the coefficient of variation is correspondingly reduced. The data obtained can be analyzed by statistical methods to determine the limits of uncertainty to be applied.

11.2.7 *Spacing Factor* $(\bar{L})$:

11.2.7.1 When r is equal to or less than 4.342 use Eq 12.

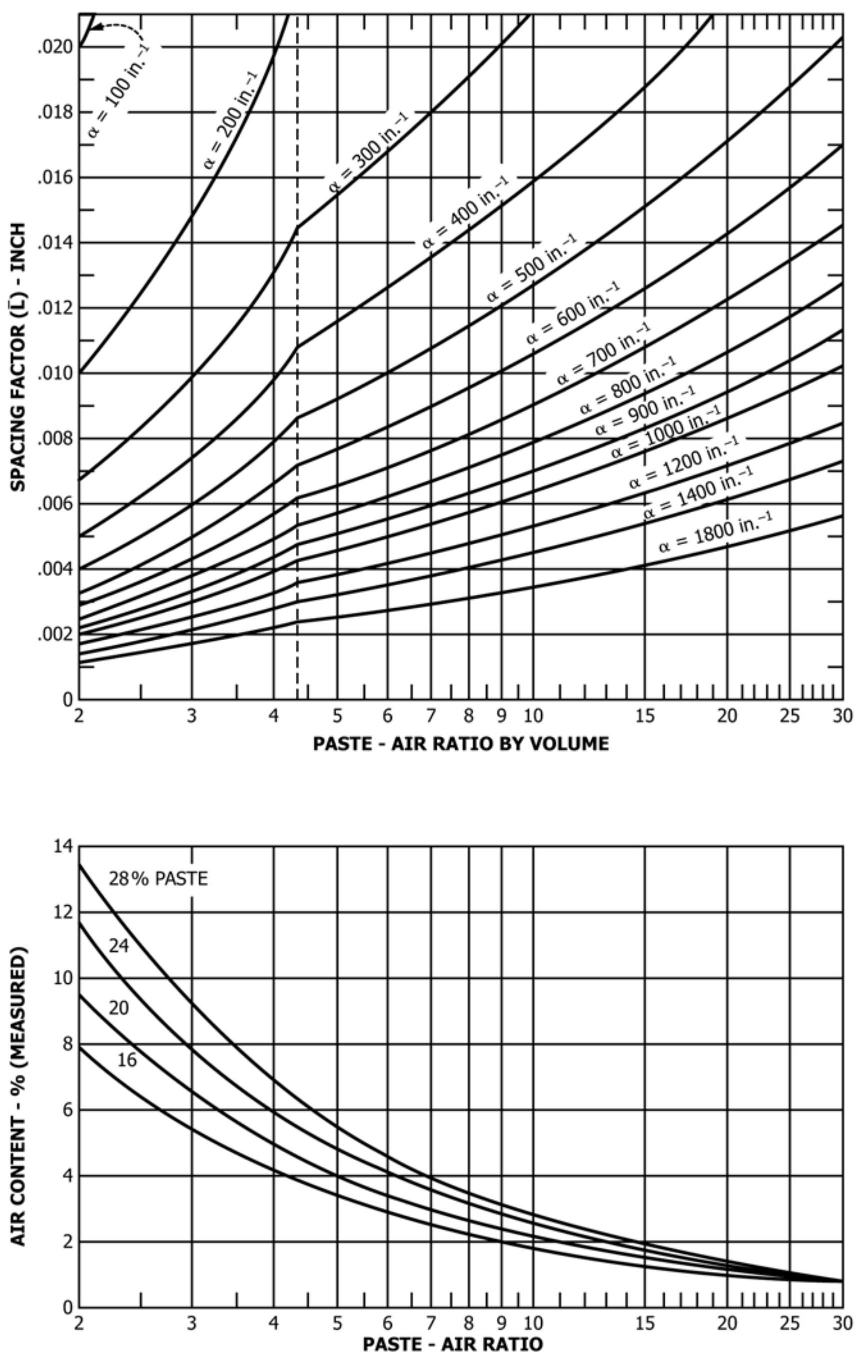
11.2.7.2 When r is greater than 4.342 use Eq 13 with the substitution of r for the *p/A* ratio.

11.3 If desired, the spacing factor $(\bar{L})$ may be estimated graphically by use of Fig. 4 rather than calculated directly.

PROCEDURE B—MODIFIED POINT-COUNT METHOD

12. Apparatus for Measurement of Specimens

12.1 The apparatus listed in 12.1.1 to 12.1.5 comprises a recommended minimum selection. Apparatus other than that described has been used and may be equally satisfactory.



NOTE 1—Estimate the spacing factor; $\bar{L}$, as follows:

(*1*) If air content was measured, selected appropriate value in lower diagram, follow horizontally to calculated or estimated paste content. Now move vertically to upper diagram to calculated specific surface, α, and then horizontally to corresponding spacing factor, $\bar{L}$, or

(*2*) If paste-air ratio was measured, select the appropriate value in the upper diagram, move vertically upward to calculated specific surface, α, and then horizontally to corresponding factor, $\bar{L}$

FIG. 4 Graphs for Estimating the Spacing Factor, $\bar{L}$

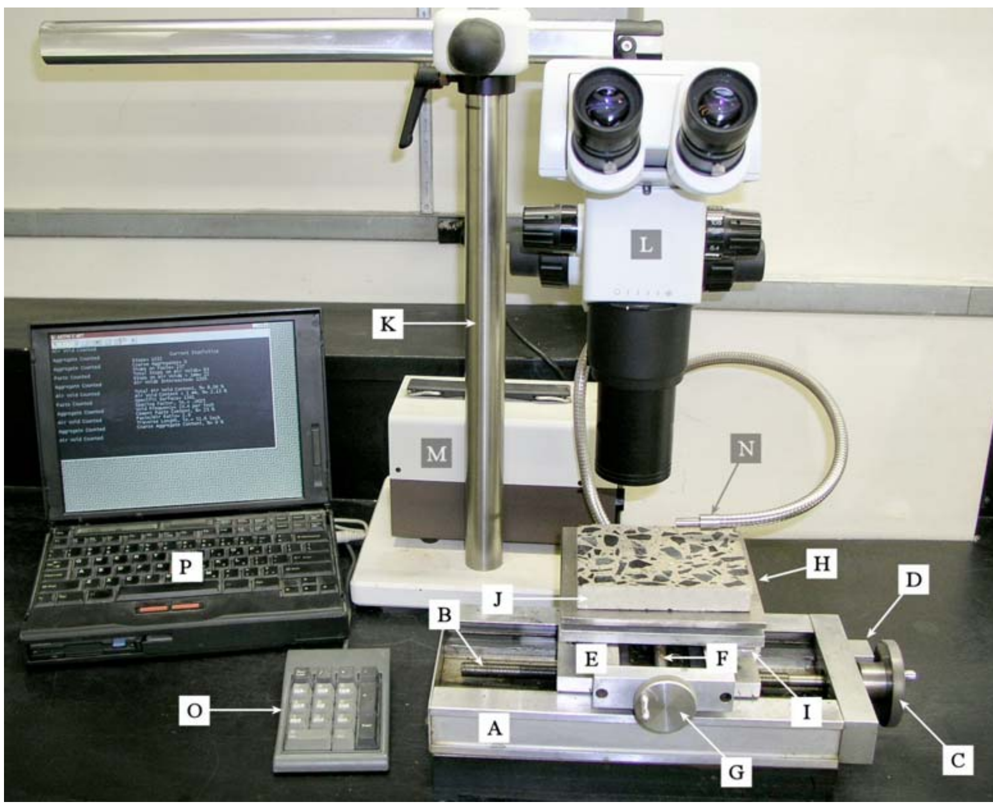
Apparatus that uses electronic switches and totalizers has been constructed. Computerized apparatus is commercially available. Image analyzers have frequently been used.

12.1.1 *Point-Count Device,* comprising a stage or platform connected to E-W and N-S lead screws (see 9.1.1) and designed in such a way that a specimen of concrete placed on the stage can be moved smoothly and uniformly through equal distances by turning of the screws. Ensure that the total possible translation of the stage is at least 100 mm [4.0 in.] in each direction. Fit lead screws with notched wheels and stopping devices, such that with each rotation of the screws a click can be detected by the operator when a stop position is reached. Ensure that the intervals between the stops correspond to a translation of the stage a distance of 0.6 to 5.0 mm [0.025 to 0.200 in.]. Determine the magnitude of the average translation of the stage between stops to the nearest 0.025 mm [0.001 in.]. Provide at least four digital counters; more may be better. It may be convenient to attach one counter to the stopping device of the E-W lead screw, so as to register automatically the total number of stops in that direction. A photograph of a satisfactory device for the modified point-count method is given in Fig. 5.

12.1.2 *Stereoscopic Microscope and Support,* as described in 9.1.2.

12.1.3 *Microscope Lamp,* as described in 9.1.3.

12.1.4 *Spirit Level,* as described in 9.1.4.



A = Lower stage assembly
B = Lower lead screw (E-W)
C = Manually operated crank for turning lower lead screw
D = Stopping device for indicating point-count positions on the line of traverse
E = Upper stage assembly
F = Cross feed screw for lateral movement of upper stage (N-S)
G = Manually operated knurled knob for turning cross feed screw
H = Plate supporting concrete specimen
I = Screws for leveling surface of concrete specimen
J = Concrete specimen
K = Support for microscope
L = Stereoscopic microscope
M = Fiber optic light source
N = Fiber optic light guide
O = Keypad to enter number of points counted, points superimposed on sections of air voids, cement paste, and the number of air-void sections intercepted by the line of traverse
P = Computer for calculations and storage of results

FIG. 5 Photograph of a Point-Count Device Meeting the Requirements of This Test Method

12.1.5 *Leveling Device,* as described in 9.1.5.

13. Procedure

13.1 Place the prepared surface of concrete on the stage of the point-count device. Using the spirit level, level the prepared surface with the leveling device so that the surface may be traversed and microscopically examined with a minimum of refocusing. Adjust the lamp so the beam evenly illuminates the field of view of the microscope and is incident upon the surface at a low angle, so the air voids are demarked by a shadow. Superimpose the index point of the cross hairs (or other reticle device) on the surface to be examined. Use a magnification not less than 50× and do not change it during the course of the analysis. For a rectangular section, place the index near an upper corner; for a circular section, place it near the top and at one end of the initial traverse. Position the stopping device at a stop or click position at the beginning of the traverse. Do not include the initial stops for each traverse line in the total number of stops or in the number of stops for any component. Zero all counters. By operation of the E-W lead screw, cause movement of the stage and specimen while simultaneously scrutinizing the surface. At each click stop, except not at the beginning of any traverse line, pause and examine the field of view, and record on the appropriate counter the material or phase on which the index point is superimposed.

NOTE 12—Surfaces examined may exhibit features that resemble air voids, but are not: *(a)* Occasionally a transparent section of a grain of quartz sand will look like an air void. *(b)* The socket left when a section of a nearly spherical and smooth sand grain is lost from the surface during grinding looks much like an air void, but can be distinguished by differences in the luster and sheen of the film lining the hole. *(c)* Cenospheres, hollow particles of fly ash, or hollow plastic spheres will also have a different sheen, and are unaffected when the surrounding paste is etched with dilute (10 %) hydrochloric acid.

Warning—Do not acid-etch the specimen under the microscope, as the effervescent spatter may damage the lens.

NOTE 13—Occasionally, air voids may become filled, during the service exposure of the concrete, with secondary products. Whether such voids are counted as belonging to the air-void system or not depends on the purposes of the investigation.

Normally use one counter for air voids, one for paste, and one for all other phases (or a totaling counter). Other components (fine and coarse aggregate, for example—if they are lithologically distinguishable) of the concrete can be determined with the use of additional counters. Continue in this way along the line until a last stop is reached just within the prepared area, but close to its edge. When the end of the line is reached, turn off the totaling counter. Reverse the E-W lead screw and proceed back along the same line, recording on another counter each air void intersected, whether or not a stop occurred within the air void. Terminate the void counting just before the initial stop. Counting of the air voids intersected during the same traverse used for counting the phases at each click stop is not prohibited. Take extreme care to determine whether a section of an air void is intersected by the movement of the index when the line of traverse is nearly tangent to the void section. The results can be affected significantly by consistent error in this respect. If the periphery of an air void is crumbled or rounded, estimate the position of the true periphery in the plane of the surface by extrapolation of the surface contour of the air void. If the examination is being made to determine only the air content of the concrete, the number of air voids intersected by the line of traverse need not be determined. By means of the N-S lead screw, shift the concrete specimen at right angles to the direction of traverse an appropriate distance. Space the segments of the traverse so as to cover the whole prepared surface and achieve at least the minimum length of traverse and the minimum number of points specified in Table 3. Proceed along the new line of traverse as before, and so on, for all segments of the total traverse and for all sections prepared from a specimen of concrete so as to comply with the requirements of this test method. Electronic or computerized equipment will require that the procedures specified by the fabricator be followed but the principles will remain as detailed above. When calculations are based on the paste-air ratio, the minimum length of traverse shall be as shown in Table 3, and the minimum number of points shall comply with the following equation:

$$S_a = \frac{10\,000}{V^2}$$

where:

S_a = number of stops at which the index point is superimposed on a section of an air void, and
V = the desired coefficient of variation of the air content expressed as a percent of the average air content.

TABLE 3 Minimum Length of Traverse and Minimum Number of Points for the Modified Point-Count Method[A]

Nominal or Observed Maximum Size of Aggregate in the Concrete, mm [in.]	Length of Traverse for Determination of A, α, or $\bar{L}$, min, mm [in.]	Number of Points for Determination of A, α, or $\bar{L}$, min
150 [6]	4064 [160]	2400
75 [3]	3048 [120]	1800
37.5 [1½]	2540 [100]	1500
25.0 [1]	2413 [95]	1425
19.0 [¾]	2286 [90]	1350
12.5 [½]	2032 [80]	1200
9.5 [⅜]	1905 [75]	1125
4.25 (No. 4)	1397 [55]	1000

[A] The limits of uncertainty of results obtained for air-void content depend upon the number of points and the air-void content of the concrete. The recommended minimum number of points shown in this table should produce limits of uncertainty such that up to 3 % air-void content the standard deviation is not greater than 0.5 % which at 3 % air-void content corresponds to a coefficient of variation of 17 %. For number of points greater than 1000 and air-void contents greater than 3 % the coefficient of variation is correspondingly reduced. The data obtained can be analyzed by statistical methods to determine the limits of uncertainty to be applied.

14. Calculation

14.1 When based on data collected from a specimen of the total concrete; the data will consist of:

where:

N = total number of air voids intersected,
S_t = total number of stops,
S_a = number of stops in air voids,
S_p = number of stops in paste (if determined), and
I = the E-W translation distance between stops.

NOTE 14—S_i, etc. may be used to denote other constituents tallied on other counters.

14.1.1 Calculate the *total traverse length* (T_t):

$$T_t = S_t \cdot I \tag{20}$$

14.1.2 *Air Content (A):*

$$A = \frac{S_a \cdot 100}{S_t} \tag{21}$$

14.1.3 *Void Frequency (n):*

$$n = \frac{N}{T_t} \tag{22}$$

14.1.4 *Paste Content* (*p*)in %:

$$p = \frac{S_p \cdot 100}{S_t} \tag{23}$$

14.1.5 *Paste-Air ratio (p/A):*

$$\frac{p}{A} = \frac{S_p}{S_a} \tag{24}$$

14.1.6 *Average chord length* $(\bar{l})$:

$$\bar{l} = \frac{S_a \cdot I}{N} \tag{25}$$

or

$$\bar{l} = \frac{A}{100n} \tag{26}$$

14.1.7 *Specific Surface* (α):

$$\alpha = \frac{4}{\bar{l}} \tag{27}$$

or

$$\alpha = \frac{400n}{A} \tag{28}$$

14.1.8 *Spacing Factor* $(\bar{L})$:

14.1.8.1 If *p/A* is less than or equal to

$$4.342\ \bar{L} = \frac{p}{400n} \tag{29}$$

14.1.8.2 If *p/A* is greater than

$$4.342\ \bar{L} = \frac{3}{\alpha}\left[1.4\left(1+\frac{p}{A}\right)^{1/3} - 1\right] \tag{30}$$

14.2 If the calculations are based on the paste-air ratio method, the data will consist of S_a , S_p, *N,* and *I* (the distance between stops). Calculate the following:

14.2.1 The *paste-air ratio (r):*

$$r = \frac{S_p}{S_a} \tag{31}$$

14.2.2 From the mixture design, calculate the ratio of *aggregate volume to paste volume (M):*

$$M = \frac{G_c}{p_c} \tag{32}$$

where:

G_c = sum of the masses of the aggregates; each divided by its specific gravity), and,
p_c = sum of mass of cement divided by specific gravity of cement + mass of mineral admixture divided by the specific gravity of the mineral admixture + mass of water (including the fluid portion of any admixture) divided by the specific gravity of the fluids.

NOTE 15—The values used for the calculated G_c and p_c must accurately reflect the differing densities of the constituents and the proportions of the masses of each.

14.2.3 The *percent air (A):*

$$A = \frac{100}{r(1+M)+1} \tag{33}$$

14.2.4 *Paste* expressed as percent *(p):*

$$p = A \cdot r \tag{34}$$

14.2.5 *Specific Surface* (α):

$$\alpha = \frac{4N}{S_a \cdot I} \tag{35}$$

14.2.6 *Average chord length* $(\bar{l})$:

$$\bar{l} = \frac{S_a \cdot I}{N} \tag{36}$$

14.2.7 *Spacing Factor* $(\bar{L})$:

14.2.7.1 When *r* is equal to or less than

$$4.342\ \bar{L} = \frac{S_p \cdot I}{4N} \tag{37}$$

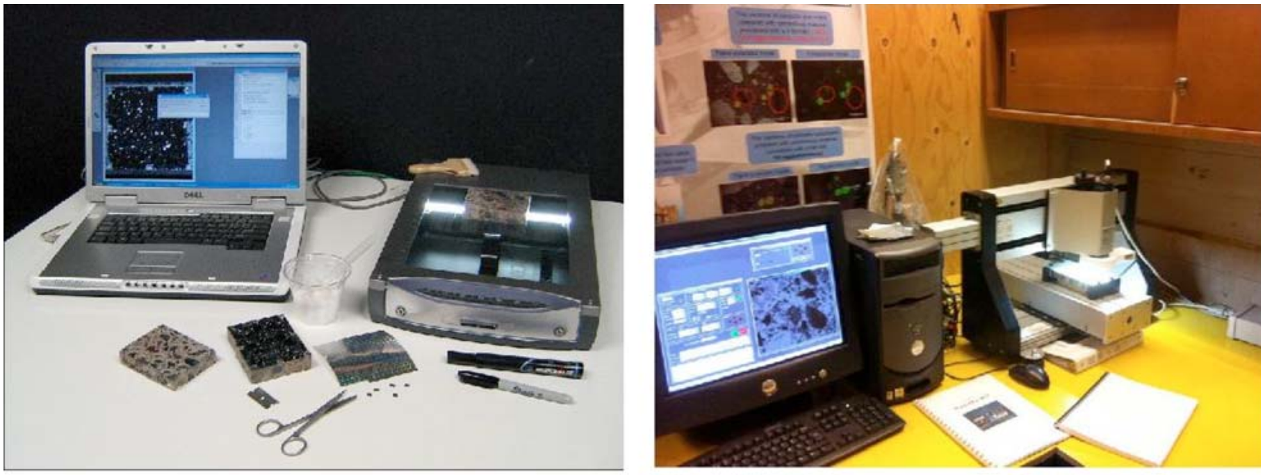
14.2.7.2 When *r* is greater than 4.342, use Eq 12 with the substitution of *r* for the *p/A* ratio.

14.3 If desired, the spacing factor *(L)* may be estimated graphically by use of Fig. 4 rather than calculated directly.

PROCEDURE C—CONTRAST ENHANCED METHOD

15. Apparatus for Measurement of Specimens

15.1 The apparatus to accomplish this procedure can vary and that listed in 15.1.1 to 15.1.5 represents the necessary requirements. Apparatus other than that described can be used and be equally satisfactory. Computerized apparatus is commercially available. Image analyzers have frequently been used. Photographs of example devices for the contrast-enhanced method are given in Fig. 6



A B

A = Device based on flat bed scanner.
B = Commercially available device based on translation of a fixed-focus optical microscope.

FIG. 6 Photographs of Two Different Devices for Performing the Contrast Enhanced Method

15.1.1 *Specimen Stage*—Provide a platform to support the specimen with the surface to be analyzed remaining perpendicular to the optical axis of the image capture device.

15.1.1.1 If the specimen must be leveled, steps described in 9.1.4 and 9.1.5 may be followed.

15.1.1.2 If the specimen is supported with the surface to be analyzed against a glass plate, with the image capture device on the opposite side of the glass plate, then place thin spacers (i.e., 0.1 mm [0.004 in.] or less) between the specimen and the glass plate to protect both surfaces from abrasion.

15.1.1.3 If it is required to translate the specimen above or beneath the image capture device, the stage shall have a minimum travel sufficient to allow for analyzing a total traverse length meeting the requirements of Table 2 without repositioning the specimen during the analysis. All movement shall occur in the focal plane of the image capture device.

15.1.1.4 If it is required to translate the image capture device above or beneath the specimen, the image capture device shall have a minimum travel sufficient to allow for analyzing a total traverse length meeting the requirements of Table 2 without repositioning the specimen during the analysis. All movement shall occur in the focal plane of the image capture device.

15.1.2 *Image Capture Device*—Provide a means to obtain the necessary digital image from the specimen. Depending on the system configuration a single image, or multiple images combined, can be used to represent the entire analysis area.

15.1.2.1 The raw data (i.e., line length) from the digital image will be in units of number of pixels. The pixel dimension (R) in the direction of measurement must be provided by the manufacturer, or established by the operator, prior to analysis to express line lengths in units of micrometers (µm) or millimeters (mm). If R is a constant for the image capture device, it need only be established once. If R is variable (i.e., the image capture device does not have a fixed magnification) then R must be established for each analysis. This is shall be established using a known calibration standard.

15.1.3 *Illumination Source*—The illumination source will vary with the image capture device configuration. In general, it must provide sufficient light to illuminate the white-powder-filled voids while not providing excess illumination that may affect the ability of the image capture device to detect the edge of an air void.

15.1.4 *Image Storage Device*—The digital image from the specimen may be stored on either a storage medium such as a disk drive, or in memory within the image processing device, until all analyses are completed.

15.1.5 *Image Processing Device*—Provide a means to perform the necessary processing steps on the digital image from the specimen including converting pixel dimensions into length using R, threshold application to segment the image, and determination of the total length traversed (T_t), the length traversed through air voids (T_a), and the number of air voids intersected by the traverse line *(N)*.

NOTE 16—Surfaces examined may exhibit features that resemble air voids, but are not: *(a)* The socket left when an aggregate grain is lost from the surface (pullout) during specimen preparation may be detected as an air void. Examine the surface for white powder- filled voids that are not circular. Either darken detected aggregate pullouts with the black felt tip marker and re-measure the specimen, re-prepare the surface if numerous pullouts are detected and re-measure the specimen, or use image processing procedures to remove those features from the image before analyzing. *(b)* In the case of high volume fly ash (HVFA) concrete, cenospheres, hollow particles of fly ash, or hollow plastic spheres may be detected as an air void. This can affect the measured air-void system parameters and it may be necessary to analyze such concrete using Procedure A or Procedure B, depending on the purpose of the analysis.

16. Procedure

16.1 If the purposes of the analysis require paste-air ratio or spacing factor, or both, determine the specimen paste content *(p)* using the mixture design parameters or by other means such as a point count.

NOTE 17—If the paste content is to be determined by point count, it is recommended that a minimum of 500 total points be examined. The uncertainty of the paste content determination will be reduced with an increased number of total points counted.

16.2 Place the prepared specimen of concrete on the specimen stage. Level the prepared surface if required, establish the analysis area, and obtain digital image(s) from the specimen using the image capture device.

16.3 Perform the necessary processing steps on the digital image(s) from the specimen including threshold application, segmenting, and determination of the total length traversed (T_t) in millimeters (mm), the length traversed through air voids (T_a) in millimeters (mm), and the number of air voids intersected by the traverse line (N).

NOTE 18—When segmenting the image the operator forms a binary image from the acquired image data. In most cases this process results in rejection of features smaller than a specified size. The largest feature rejected while performing the thresholding operation is selected by the operator based on the apparatus being used and the purposes of the analysis.

17. Calculation

17.1 The data will consist of the following:

T_t = total length traversed, mm,
T_a = length traversed through air voids, mm,
N = number of air voids intersected by the traverse line, and
p = paste content, if determined, volume %.

17.2 Perform calculations as described in Section 11.

18. Report

18.1 Report the following information:

18.1.1 Method used, Procedure A, Procedure B, or Procedure C.

18.1.2 Identification of the source of the specimens,

18.1.3 Location from which the specimens were taken and their orientation with respect to the sources,

18.1.4 Orientation and position of the surfaces cut from the samples for traversing,

18.1.5 The length of traverse, the area traversed, and, if the modified point-count method is employed, the number of stops,

18.1.6 The determined values of air content and, if measured, paste content, void frequency, specific surface, spacing factor, and paste-air ratio,

18.1.7 Method by which the paste content was determined, and

18.1.8 Magnification used during the analysis.

18.2 If the parameters of the air-void system have been determined on near-surface sections, the relationship of such parameters to the depth from the reference surface.

19. Precision and Bias

19.1 *Precision:*

19.1.1 The estimates of precision for this test method are given in Table 4 for determinations made on a series of four lapped specimens prepared in one laboratory and then circulated to nine laboratories where a number of determinations were made in each laboratory, usually involving at least two operators per laboratory and two or three determinations per operator. The range of properties covered by the four prepared surfaces used in this study was: for air content, 2 to 9 %; for voids intersected, 79 to 512 voids/m [2 to 13 voids/in.]; and for paste content average, in the range of 20 to 22 %. The variability of the test method would be higher in actual practice for specimens obtained and prepared from in-place concrete since additional variation due to specimen selection and surface preparation in different laboratories would increase the coefficient of variation. Table 5 compares the actual variation

TABLE 4 Average Precision Data Based on a Set of Four Prepared Specimens Circulated to Nine Laboratories[A]

Item	Standard Deviation (1s)[B]	Range of Two Test Results (D2s)[B]	Percent of Average: Coefficient of Variation (1s %)[C]	Percent of Average: Range of Two Test Results (D2s %)[C]
Air Content, %:				
Within Lab	0.29	0.82	. . .	. . .
Between Lab	0.41	1.16	. . .	. . .
Voids/ cm [in.]:				
Within Lab	. . .	. . .	1.5 [3.7]	4.1 [10.3]
Between Lab	. . .	. . .	4.8 [12.4]	13.8 [35.0]
Paste Content, %:				
Between Operator (Each in Their Own Laboratory)	3.1	8.8	. . .	. . .

[A] Includes determinations using both point count and linear traverse. In all, 19 operators participated with a total of 41 determinations on each specimen. For point count: 6 labs; 12 operators; and 26 determinations. For linear traverse: 3 labs; 7 operators; and 15 determinations. Paste content determinations were made by 13 operators representing 8 different laboratories. Eleven were by point count and 2 by linear traverse procedures.
[B] These numbers represent, respectively, the (1s) and (D2s) limits as described in Practice C670.
[C] These numbers represent, respectively, the (1s %) and (D2s %) limits as described in Practice C670.

TABLE 5 Comparison of Actual Variation with Theoretical Estimates Based on Binomial and Poisson's Distribution

Specimen Number	Average Value	Standard Deviation: Actual[A]	Standard Deviation: Theoretical[B]	Coefficient of Variation %: Actual	Coefficient of Variation %: Theoretical
Air Content, % Based on Counts of 1500 Points					
1	2.98	0.45	0.44	15.1	14.8
2	3.65	0.49	0.48	13.4	13.2
3	5.55	0.40	0.59	7.2	10.6
4	8.02	0.64	0.70	8.0	8.7
Voids Per Inch Based on 100 in. Traverses					
1	2.31	0.27	0.15	11.7	6.5
2	5.77	0.78	0.24	13.5	4.2
3	7.69	0.86	0.28	11.2	3.6
4	12.69	1.63	0.36	12.8	2.8

[A] Includes all data—all labs and all operators. Includes both point count and linear traverse data.
[B] Theoretical Standard Deviation:—For air content: Binomial Distribution; Standard deviation in points on air $= \sqrt{npq}$ using an assumed 1500 point count, where n is number of points, p is decimal fraction of air, and q is decimal fraction not-on-air. To convert to units of percent air content, divide by n and multiply by 100 to get percent. For voids per in.: Poisson's Distribution; $S.D. = \sqrt{np}$ where np is the mean number of occurrences (counts per 2540 [100 in.] of traverse based on results of 2540–mm [100-in.] traverses).

from this study with theoretical estimates.

19.1.2 Table 6 provides an estimate of precision for air content and spacing factor from a European study[4] using predominantly the same procedures as contained in this test method. The great majority of these determinations were made by linear traverse with a magnification of 50×. Two hardened concrete beam specimens with nominal air contents of about 3 and 6.5 % were obtained by one laboratory. A pair of surfaces were sawn from the beams for each participating laboratory; one surface was lapped in the originating laboratory, and the adjacent surface was lapped in the participating laboratory.

19.1.3 The repeatability standard deviation for Procedure C is provided in Table 7. The reproducibility (interlaboratory standard deviation) of Procedure C has not yet been determined.

19.2 *Bias:*

19.2.1 There is no accepted reference material suitable for determining bias from the true air-void parameters of concrete.

19.2.2 From the results of the interlaboratory study referenced in 19.1.1 and Table 4 there was very little bias between Procedure A and Procedure B for the average results for air content and voids intersected measured on each of the four specimens. The overall averages are given in Table 8. Too few determinations of paste content were made by Procedure A to make any comparison for that property.

[4] Sommer, Hermann, "The Precision of the Microscopical Determination of the Air-Void System in Hardened Concrete," *ASTM, Cement, Concrete, and Aggregate*, Vol 1, No. 2, 1979, pp. 49–55.

TABLE 6 Estimate of Average Precision from a European Study Where Both Prepared Specimens and Companion Unprepared Specimens Were Sent to Participating Laboratories to Determine Air Content and Spacing Factor

Item	Standard Deviation (1s)[A]	Range of Two Test Results (D2s)[A]	Percent of Average	
			Coefficient of Variation (1s %)[B]	Range of Two Test Results (D2s %)[B]
Slabs Prepared in Originating Lab and Measured in Originating Lab				
Air Content, %	0.57	1.61	. . .	. . .
Spacing Factor	. . .	. . .	8.0	22.6
Slabs Prepared in Originating Lab But Measured in Participating Labs				
Air Content, %	0.71	2.01	. . .	. . .
Spacing Factor	. . .	. . .	20.1	56.9
Slabs Lapped and Measured in Participating Labs				
Air Content, %	0.73	2.07	. . .	. . .
Spacing Factor	. . .	. . .	17.5	49.5

[A] These numbers represent, respectively, the (1s) and (D2s) limits as described in Practice C670.

[B] These numbers represent, respectively, the (1s %) and (D2s %) limits as described in Practice C670.

20. Keywords

20.1 air content; air void parameters of hardened concrete; area-prepared surface required; average chord-length of voids; determination of air-void parameters; entrained void; entrapped void; grits for lapping hardened concrete; index point; lapping; length of traverse required; linear-traverse method; microscopical; number of points required; paste-air ratio; paste-air ratio modification; paste content; point-count method (modified); specimen preparation for microscopical analysis; spacing factor; specific surface; void frequency; water void

TABLE 7 Repeatability Standard Deviation for Procedure C Performed Using Two Different Devices

Specimen[A]	Air Content, %		Voids/cm [in.$^{-1}$]		Specimen[D]	Air Content, %		Voids/cm [in.$^{-1}$]	
	Device A[B]	Device B[C]	Device A[B]	Device B[C]		Device A[B]	Device B[C]	Device A[B]	Device B[C]
Analysis 1	4.90	4.65	2.88 [7.3]	2.78 [7.1]	Analysis 1	6.66	6.94	4.65 [11.8]	5.06 [12.9]
Analysis 2	4.98	4.52	2.90 [7.4]	2.65 [6.7]	Analysis 2	6.75	6.94	4.72 [12.0]	5.12 [13.0]
Analysis 3	4.81	4.53	2.80 [7.1]	2.64 [6.7]	Analysis 3	6.70	6.95	4.69 [11.9]	5.08 [12.9]
Analysis 4	4.85	4.53	2.77 [7.0]	2.67 [6.8]	Analysis 4	6.81	6.86	4.72 [12.0]	4.91 [12.5]
Analysis 5	4.7	4.52	2.73 [6.9]	2.65 [6.7]	Analysis 5	6.78	6.88	4.66 [11.8]	4.92 [12.5]
Average (n = 5)	4.85	4.55	2.82 [7.2]	2.68 [6.8]	Average (n = 5)	6.74	6.91	4.69 [11.9]	5.02 [12.7]
Standard Deviation	0.09	0.06	0.07 [0.2]	0.06 [0.2]	Standard Deviation	0.06	0.04	0.03 [0.1]	0.10 [0.3]
C of *V*	1.9 %	1.2 %	2.6 %	2.2 %	*C* of *V*	0.9 %	0.6 %	0.7 %	1.9 %
Specimen[A]	Spacing Factor, mm [in.]		Specific Surface, mm^{-1} [in.$^{-1}$]		Specimen[D]	Spacing Factor, mm [in.]		Specific Surface, mm^{-1} [in.$^{-1}$]	
	Device A[B]	Device B[C]	Device A[B]	Device B[C]		Device A[B]	Device B[C]	Device A[B]	Device B[C]
Analysis 1	0.215 [0.008]	0.216 [0.009]	23.5 [596.8]	23.9 [607.7]	Analysis 1	0.157 [0.006]	0.147 [0.006]	27.9 [709.9]	29.2 [741.4]
Analysis 2	0.215 [0.008]	0.216 [0.009]	23.3 [592.0]	23.4 [594.5]	Analysis 2	0.156 [0.006]	0.145 [0.006]	27.9 [709.8]	29.5 [749.3]
Analysis 3	0.219 [0.009]	0.226 [0.009]	23.3 [591.1]	23.3 [591.8]	Analysis 3	0.156 [0.006]	0.147 [0.006]	28.0 [711.6]	29.2 [742.5]
Analysis 4	0.222 [0.009]	0.221 [0.009]	22.9 [581.5]	23.6 [600.0]	Analysis 4	0.157 [0.006]	0.152 [0.006]	27.7 [703.3]	28.6 [727.2]
Analysis 5	0.223 [0.009]	0.224 [0.009]	23.0 [583.9]	23.4 [594.9]	Analysis 5	0.159 [0.006]	0.150 [0.006]	27.5 [698.6]	28.6 [726.7]
Average (n = 5)	0.219 [0.009]	0.221 [0.009]	23.2 [589.1]	23.5 [597.8]	Average (n = 5)	0.157 [0.006]	0.148 [0.006]	27.8 [706.6]	29.0 [737.4]
Standard Deviation	0.004 [0.000]	0.005 [0.000]	0.25 [6.3]	0.25 [6.3]	Standard Deviation	0.001 [0.000]	0.003 [0.000]	0.22 [5.5]	0.39 [10.0]
C of *V*	1.7 %	2.1 %	1.1 %	1.1 %	*C* of *V*	0.8 %	1.9 %	0.8 %	1.4 %

[A] The same concrete specimens were analyzed by both devices. The nominal concrete mixture design was 222 kg [489 lb] cement, 55.5 kg [122 lb] Class C fly ash, 833 kg [1836 lb] coarse aggregate (dolomitic limestone; absorption 0.7 %), 542.5 kg [1196 lb] fine aggregate (natural sand; absorption 0.5 %), w/c = 0.45, paste content 29.7 %, wood rosin air entraining agent, and a target air content of 5 %. Using each device, one specimen was analyzed five times in repetition. After each analysis, the specimen was removed from the device and then replaced on the device to capture the next analysis.

[B] Device conforming to the specifications described herein for Procedure C. Example device shown in Fig. 6A. Measurements were made at the University of Toronto, Toronto, Ontario, Canada, by a single operator.

[C] Device conforming to the specifications described herein for Procedure C. Example device shown in Fig. 6B. Measurements were made at the Oklahoma State University, Stillwater, Oklahoma, United States, by a single operator.

[D] The same concrete specimens were analyzed by both devices. The nominal concrete mixture design was 222 kg [489 lb] cement, 55.5 kg [122 lb] Class C fly ash, 833 kg [1836 lb] coarse aggregate (dolomitic limestone; absorption 0.7 %), 542.5 kg [1196 lb] fine aggregate (natural sand; absorption 0.5 %), w/c = 0.45, paste content 29.7 %, wood rosin air entraining agent, and a target air content of 7 %. Using each device, one specimen was analyzed five times in repetition. After each analysis, the specimen was removed from the device and then replaced on the device to capture the next analysis.

TABLE 8 Comparison of Overall Average Data on the Set of Four Prepared Specimens Circulated to Different Laboratories and Operators

Specimen	Air Content, %		Voids/cm [in.$^{-1}$]	
	Point Count[A]	Linear Tr.[B]	Point Count[A]	Linear Tr.[B]
No. 1	3.00	2.91	0.88 [2.24]	0.92 [2.33]
No. 2	3.70	3.59	2.24 [5.69]	2.12 [5.39]
No. 3	5.66	5.46	2.93 [7.44]	2.93 [7.44]
No. 4	8.20	7.97	4.89 [12.42]	4.79 [12.17]

[A] Modified Point Count: 6 labs; 12 operators; and 26 determinations.

[B] Linear Traverse: 3 labs; 7 operators; and 15 determinations.

APPENDIX

(Nonmandatory Information)

X1. INTERPRETATION OF RESULTS

X1.1 The question of whether or not the determined air-void system parameters are acceptable to provide protection in water-saturated cyclic freezing and thaw conditions shall be made by the specifier, or be determined from actual freeze-thaw testing of the concrete (that is, Test Method C666/C666M, Test Method C672/C672M, field results). The following text provides useful information on air-void system parameters of known freeze-thaw durable concrete and explains guidelines and specifications from other respected specifying agencies.

X1.2 For air-entrained concrete designed in accordance with ACI 201.2R and ACI 211.1, the paste-air ratio *(p/A)* is usually in the range 4 to 10, the specific surface (α) is usually in the range 25 to 45 mm^{-1} [600 to 1100 $in.^{-1}$], and the spacing factor $(\bar{L})$ is usually in the range 0.1 to 0.2 mm [0.004 to 0.008 in.].

X1.3 Of the parameters determined with this test method, the spacing factor $(\bar{L})$ is generally regarded as the most significant indicator of the durability of the cement paste matrix to freezing and thawing exposure of the concrete. The maximum value of the spacing factor for moderate exposure of the concrete is usually taken to be 0.20 mm [0.008 in.]. Somewhat larger values may be adequate for mild exposure, and smaller ones may be required for severe exposure, especially if the concrete is in contact with deicing chemicals. Care should be exercised in using spacing factor values in specifications since the standard deviation of that property has been found to approach one-fifth of the average when determinations are made in different laboratories. Hence, substantial differences in spacing factor may be caused solely by sampling and between laboratory variation. The factors affecting the variability of the test method are discussed in the section on Precision and Bias.

X1.4 The air content and the parameters of the air-void system in hardened concrete depend primarily on the kind and dosage of the air entraining agent used, the degree of consolidation of the concrete, and its water-cement ratio. The values of the specific surface (α) and the void frequency *(n)* decrease rapidly with an increase of the water-cement ratio or the paste content if other conditions are not altered. Satisfactory values of specific surface (α) and spacing factor $(\bar{L})$ require that the void frequency be larger than about 300/m [8/in.]. An increase in the water-cement ratio or the paste content must be accompanied by an increase in the air content, if the spacing factor $(\bar{L})$ is not to increase. The air content can be reduced substantially by extended vibration of the concrete, without a significant increase of the spacing factor $(\bar{L})$, provided the concrete was adequately air entrained originally. Extended vibration is not, however, recommended as a field practice because of the dangers of excessive bleeding and segregation.

X1.5 The void frequency *(n)* is a critical parameter in determining the magnitude of the specific surface (α) and the spacing factor $(\bar{L})$. Consequently, utmost care must be taken in conducting either microscopical method to observe and record all air-void sections intersected by the line of traverse. Recognition of air-void sections of small size, for example, 10 µm [0.0004 in.] is essential to securing a correct evaluation of these parameters. For this reason, care must be taken to prepare extremely smooth and plane sections, the magnification employed should be not less than 50×, and the index point in the cross hairs (or other reticle device) must be observed precisely in relation to the area and periphery of the air-void section.

X1.6 Provided the value of the specific surface (α) or the void frequency *(n)* is sufficiently high, a suitable spacing factor $(\bar{L})$ will be obtained even when the air content is low. However, in order to obtain an air-void system that has both the volume capacity and the geometric parameters necessary to protect saturated mature cement paste during exposure to freezing, it is important to obtain concrete with an acceptably high air content *(A)* and a low enough spacing factor $(\bar{L})$ to provide protection.

X1.7 For concrete exposed to freezing and thawing while critically saturated, a minimum-compressive strength must be developed prior to the freezing exposure, in addition to the securing of adequate air entrainment if the concrete is to be protected properly. Such compressive strength must be at least 28 MPa [4000 psi].

SUMMARY OF CHANGES

Committee C09 has identified the location of selected changes to this test method since the last issue, C457/C457M–12, that may impact the use of this test method. (Approved December 1, 2016.)

(1) Added Procedure C.