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Standard Specification for Ground Calcium Carbonate and Aggregate Mineral Fillers for use in Hydraulic Cement Concrete[1]

This standard is issued under the fixed designation C1797M; 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 specification applies to ground calcium carbonate (GCC is a type of ground limestone) and other finely divided aggregate mineral filler (AMF) materials for use in concrete mixtures. The specification defines the types of GCC and AMF materials for use in concrete.

1.2 If concrete in service is subject to sulfate exposure, fillers derived from ground limestone should not be used unless mitigation methods are used.

NOTE 1—American Concrete Institute (ACI) technical documents 201.2R, 318, 332, and 350 contain useful information and code requirements dealing with sulfate exposure in service. Soluble sulfate in water can be determined in accordance with Test Method D516 or Test Method D4130. Percent sulfate by mass in soil can be determined by Test Method C1580.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

NOTE 2—Sieve size is identified by its standard designation in Specification E11. The alternative designation given in parentheses is for information only and does not represent a different standard sieve size.

1.4 The text of this standard references notes and footnotes, which provide explanatory information. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C25 Test Methods for Chemical Analysis of Limestone, Quicklime, and Hydrated Lime
C50/C50M Practice for Sampling, Sample Preparation, Packaging, and Marking of Lime and Limestone Products
C51 Terminology Relating to Lime and Limestone (as used by the Industry)
C110 Test Methods for Physical Testing of Quicklime, Hydrated Lime, and Limestone
C117 Test Method for Materials Finer than 75-μm (No. 200) Sieve in Mineral Aggregates by Washing
C125 Terminology Relating to Concrete and Concrete Aggregates
C136/C136M Test Method for Sieve Analysis of Fine and Coarse Aggregates
C311/C311M Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete
C566 Test Method for Total Evaporable Moisture Content of Aggregate by Drying
C595/C595M-15 Specification for Blended Hydraulic Cements
C1580 Test Method for Water-Soluble Sulfate in Soil
C1777 Test Method for Rapid Determination of the Methylene Blue Value for Fine Aggregate or Mineral Filler Using a Colorimeter
D75/D75M Practice for Sampling Aggregates
D516 Test Method for Sulfate Ion in Water
D1193 Specification for Reagent Water
D4130 Test Method for Sulfate Ion in Brackish Water, Seawater, and Brines
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
E832 Specification for Laboratory Filter Papers

2.2 *ACI Documents:*[3]

ACI 201.2R Guide to Durable Concrete
ACI 318 Building Code Requirements for Structural Concrete and Commentary
ACI 332 Residential Code Requirements for Structural Concrete and Commentary

[1] This specification is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.20 on Normal Weight Aggregates.

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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), 38800 Country Club Dr., Farmington Hills, MI 48331-3439, http://www.concrete.org.

ACI 350 Code Requirements for Environmental Engineering Concrete Structures and Commentary
ACI CT-13 Concrete Terminology

2.3 *Standards of Other Organizations:*

AASHTO T 330 Method of Test for the Quantitative Detection of Harmful Clays of the Smectite Group in Aggregates Using Methylene Blue[4]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this specification, refer to Terminology C51 and Terminology C125.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aggregate mineral filler (AMF), n*—a finely divided inorganic material derived from quarried stone, for use as an ingredient in hydraulic cementitious mixtures and meeting specified chemical and physical requirements.

3.2.1.1 *Discussion*—AMF derived from carbonate or non-carbonate quarried stone are finely divided particulate matter that have been shown to be effective in improving the particle packing and rheological characteristics of fresh concrete. In some cases, enhancement is seen in mechanical and fluid transport properties of hardened concrete. AMF for use in concrete may undergo a series of processing steps such as screening, grinding, classifying and drying as needed to meet requirements of this specification.

3.2.2 *ground calcium carbonate (GCC), n*—a finely divided inorganic material consisting predominantly of calcium carbonate or of the carbonates of calcium and magnesium and meeting specified chemical and physical requirements.

3.2.2.1 *Discussion*—The series of processing steps like grinding and classifying that these products undergo, ensure consistent particle size distribution. Research has demonstrated that the use of GCC results in improved packing density and the GCC particles provide nucleation sites that increase the rate of hydration of hydraulic cementitious materials. The effect of hydration is also influenced by the particle size distribution (PSD) and fineness of the GCC. See cited References **(1-6)**.[5]

4. Classification

4.1 Types A and B, which are derived from calcium carbonates, are ground products from quarried stone. The chemical and physical properties shall comply with the requirements in Table 1.

4.2 Type C is typically a byproduct from the crushing of quarried stone, with mineral composition that depend on the stone from which it is derived. The chemical and physical properties shall comply with the requirements of Table 1.

4.3 The Type classification shall be stated by the supplier of the product.

NOTE 3—ACI CT-13 defines mineral filler as a finely divided mineral product at least 65% of which passes the 75-µm (No. 200) sieve. This specification establishes requirements for GCC and AMF materials that meet this definition.

TABLE 1 Chemical and Physical Requirements

Parameter	Type A	Type B	Type C
$CaCO_3$, % by mass	≥ 92	≥ 70	NA
Sum of $CaCO_3$ + $MgCO_3$, % by mass	≥ 95	≥ 90	NA
Methylene blue value (mg/g)	≤ 3	≤ 5	≤ 5
Total Organic Carbon Content % by mass	≤ 0.5	≤ 0.5	≤ 0.5
Particle size distribution, minimum % by mass passing			
300-µm (No. 50) sieve	100	100	100
150-µm (No.100) sieve	100	85	
75-µm (No. 200) sieve	95	70	65
45-µm (No. 325) sieve	90	65	
Fineness (m^2/kg)[D]	Report[A]	Report[A]	Report[A]
Moisture Content (%)[B] by mass	≤ 1	≤ 1	≤ 1
Strength Activity Index, % of control at 28d[C]	≥ 75	≥ 75	≥ 75
Water Requirement, maximum % by mass of control	120	120	120

[A] The purchaser has the authority to approve a change in the fineness or to add a range if needed.
[B] The moisture content is listed for materials that can be pneumatically transferred. If material is not pneumatically transferred, then the purchaser can waive the moisture content requirement.
[C] The purpose of testing the Strength Activity Index is to evaluate whether the material has any detrimental effect when used in concrete.
[D] There is no specification limit but the value is reported to provide information to the purchaser. The proportioning of a concrete mixture may be dependent on the fineness of the material to be used. If there is a change in fineness, the purchaser should be notified so that appropriate adjustments can be made to the concrete mixtures.

5. General Requirements

5.1 The chemical and physical requirements for Type A, Type B, and Type C shall conform to the requirements in Table 1.

5.2 The purchaser has the authority to request measurement by a specified method of the chloride ion content of the material.

6. Sampling

6.1 Obtain a sample from each lot for testing in accordance with Practice C50/C50M or Practice D75/D75M.

7. Test Methods

7.1 Calcium carbonate and magnesium carbonate content – Test Methods C25 or Annex A1.

7.2 Methylene Blue Value – AASHTO T330 or Test Method C1777.

NOTE 4—The specification values are based on testing research using AASHTO T330 while ASTM C1777 was under development. Data comparison between AASHTO T330 and C1777 will be made when sufficient data sets become available.

7.3 Total Organic Carbon (TOC) Content – Specification C595/C595M-15 Annex A3.

7.4 Particle Size Distribution (PSD) – Test Method C110 for Types A and B. Test Method C136/C136M and Test Method C117 for Type C.

7.5 Strength Activity Index – Test Methods C311/C311M replacing the mass of pozzolan in the test mixture with GCC or AMF.

7.6 Water requirement – Test Methods C311/C311M.

[4] Available from American Association of State Highway and Transportation Officials (AASHTO), 444 N. Capitol St., NW, Suite 249, Washington, DC 20001, http://www.transportation.org.

[5] The boldface numbers in parentheses refer to the list of references at the end of this standard.

7.7 Fineness – Test Methods C110.

7.8 Moisture Content – Test Methods C25 or Test Method C566.

8. Storage and Inspection

8.1 Store the product in such a manner as to permit easy access for proper inspection and identification of each shipment.

8.2 Inspection of the material shall be made as agreed upon by the purchaser and the supplier as part of the purchase order or contract.

9. Rejection

9.1 The purchaser has the right to reject material that fails to conform to the requirements of this specification. Rejection shall be reported to the producer or supplier in writing.

10. Packaging and Package Marking

10.1 If the product is delivered in packages, the name of the manufacturer, Type, and the mass of the material contained therein shall be plainly marked on each package. Similar information shall be provided in the shipping invoices accompanying the packaged or bulk material.

11. Certification

11.1 If specified in the purchase order or contract, the purchaser shall furnish certification that samples representing each lot have been tested as directed in this specification and the specified requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

12. Manufacturer's Statement

12.1 At the request of the purchaser, the manufacturer shall state in writing the mineral origin, nature, amount, and type of any processing or other additions made to the product.

13. Keywords

13.1 aggregate mineral filler; ground calcium carbonate; hydraulic cement concrete; limestone; mineral filler

ANNEX

(Mandatory Information)

A1. CHEMICAL ANALYSIS OF CALCIUM AND MAGNESIUM CARBONATES IN GROUND LIMESTONE BY EDTA TITRATION

A1.1. Scope

A1.1.1 This test method covers the chemical analysis of high-calcium and dolomitic limestone.

A1.1.2 The standard test method uses classical gravimetric and volumetric analytical procedures and are typically required for referee analyses if conformance with chemical specification requirements is part of contractual agreement between purchaser and producer.

A1.1.3 *This standard does not purport to address all of the safety concerns, if any, 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.*

A1.2 Referenced Documents

A1.2.1 Pulverized Limestone Association Test Method 4-70

A1.3. Significance and Use

A1.3.1 This test method provides analytical procedures to determine the major chemical constituents of limestone (see Note 1). The percentages of specific constituents that determine a material's quality or fitness for use are of significance depending upon the purpose or end use of the material. Results obtained may be used in relation to specification requirements.

NOTE A1.1—This test method can be applied to other calcareous materials if provisions are made to compensate for known interferences.

A1.4. Apparatus

A1.4.1 *Balance*—The balance shall be of an analytical type with a capacity not to exceed 200 g. It may be of conventional design or it may be a constant-load, direct-reading type. It shall be capable of reproducing weighings within 0.0002 g with an accuracy of ±0.0002 g.

A1.4.2 *Glassware and Laboratory Containers*—Standard volumetric flasks, burets, pipets, dispensers, etc. should be of precision grade or better (Class A). Polyethylene containers are recommended for all aqueous solutions of alkalies and for standard solutions where the presence of dissolved silica or alkali from the glass would be objectionable.

A1.4.3 *Filter Paper*—Filter paper shall conform to the requirements of Specification E832, Type II, Quantitative, Class F.

A1.5. Reagents

A1.5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society if such specifications are available.[6] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

A1.5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I or II of Specification D1193.

A1.5.3 *Ethylenediaminetetraacetic Acid, EDTA*—Dissolve 18 g of Disodium EDTA in 1 L of water.

A1.5.4 *Potassium hydroxide, KOH*—Dissolve 56 g of KOH in 1 L of water.

A1.5.5 *Ammonia Buffer*—Dissolve 66 g of ammonium chloride, NH_4Cl, in 300 mL water in a 2 L beaker. Add 560 mL ammonium hydroxide, NH_4OH. Transfer to a 1000 mL volumetric flask and make up to volume with water.

A1.5.6 *Calcium Carbonate, $CaCO_3$*—ACS grade, 99.95 – 100.05% purity.

A1.5.7 *Dilute Hydrochloric Acid*—Dilute 1 part of concentrated HCl (12N) into 4 parts of water.

A1.5.8 *Hydroxy napthol blue indicator.*

A1.5.9 *Calmagite (1-(1-hydroxy-4-methyl-2-phenylazo)-2-napthol-4-sulfonic acid) indicator.*

A1.6 Standardization of the EDTA Titration Solution

A1.6.1 Weigh out two samples of ACS grade $CaCO_3$ into 600-mL beakers. Use 0.200 g. Record mass of each sample.

A1.6.2 Add 10 mL water and 5 mL 1+4 HCl to each beaker with $CaCO_3$. Stir solution to disperse $CaCO_3$. When $CaCO_3$ is dissolved add 285 mL water.

A1.6.3 Fill 50-mL burette with EDTA solution. To prevent precipitation of the calcium add 35 mL EDTA titrating solution. Using a graduated cylinder, add 30 mL KOH and stir. Add 0.2 g hydroxyl napthol blue indicator and stir. Complete titration to a clear blue end point by adding EDTA solution from burette with constant stirring.

A1.6.4 *Calculations:*

$$\text{Calcium carbonate factor, } Fc\text{, \%/mL} = \frac{Mc \times P}{V} \times 500 \quad (A1.1)$$

where:

Mc = mass of ACS grade calcium carbonate,
P = purity percentage of ACS grade calcium carbonate, 99.95 – 100.05, and
V = volume of EDTA to reach end point.

$$\text{Magnesium factor, } Fm\text{, \%/mL} = Fc \times 0.84254 \quad (A1.2)$$

where:
Fc = calcium carbonate factor.

A1.7. Procedure

A1.7.1 Weigh a 2.00 g limestone sample and place into a 600-mL beaker. Add 50 mL 1+4 HCl. Cover beaker with watch glass and heat to boiling point. Remove from heat when the sample goes into solution. Cool to room temperature.

NOTE A1.2—Fading end point on dolomites can be lessened by adding tartrate or a couple drops of triethanolamine.

NOTE A1.3—Interference by iron, copper or other transition elements can be obviated by addition of 10 mL of 2% KCN.

A1.7.2 Fold filter paper to fit inside glass funnel. Place funnel with filter paper into a into a 500-mL volumetric flask. Pour the prepared solution from A1.7.1 through the filter paper. Be sure to not overflow the filter paper. After all the solution is poured out, use a wash bottle of water to rinse the entire contents of the beaker into the filter paper. Wash the filter paper 5 or 6 times with enough water to fill the filter paper but does not fill the flask past the 500 mL mark on the neck of the flask. Remove funnel and filter paper. Add water to the flask to the 500-mL mark on the neck of the flask.

A1.7.3 Remove filter paper from funnel. Ignite the paper in a weighed, porcelain crucible at 750 to 900°C, cool in a desiccator, and weigh. Residue is acid insoluble material.

$$\text{Acid Insoluble, \%} = \frac{\text{Mass of residue} \times 100}{2} \quad (A1.3)$$

NOTE A1.4—The acid insoluble determination serves as a check on the determination. The % $CaCO_3$ + %$MgCO_3$ + % Acid Insoluble will be close to 100% for most limestone.

A1.7.4 Pipette two 50-mL portions into a 600-mL beaker. Add 250 mL distilled water. Add 30 mL KOH and stir.

A1.7.5 Add 0.2 grams of hydroxyl napthol blue indicator and stir. Complete titration to a blue end point by adding EDTA solution from the burette with constant stirring. Record volume of EDTA to reach endpoint.

A1.7.6 Pipette two 50-mL portions into a 600-mL beaker. Add 250 mL distilled water. Add 20 mL ammonia buffer and stir. Add same volume of EDTA solution for the calcium titration then add 0.4 grams Calmagite indicator. Complete titration to a blue end point by adding EDTA solution from the burette with constant stirring. Record the volume of EDTA to reach the endpoint.

A1.8. Calculations

$$\text{Calcium carbonate, } CaCO_3\text{, \%} = Vc \times Fc \quad (A1.4)$$

where:

Vc = volume in mL of EDTA to reach hydroxyl napthol blue titration endpoint, and
Fc = calcium carbonate factor.

$$\text{Volume of EDTA required to titrate magnesium, } Vm = Vt - Vc \quad (A1.5)$$

where:

Vt = total volume in mL of EDTA to reach Calmagite titration endpoint, and

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[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the United States *Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

Vc = volume in mL of EDTA to reach hydroxyl napthol blue titration endpoint.

$$\text{Magnesium carbonate, } MgCO_3, \% = Vm \times Fm \quad (A1.6)$$

where:

Vm = volume of EDTA required to titrate magnesium, and
Fm = magnesium carbonate factor.

A1.9. Report

A1.9.1 Report the following information to the nearest 1%:
A1.9.1.1 Calcium carbonate percentage, and
A1.9.1.2 Magnesium carbonate percentage.

A1.10. Precision and Bias

A1.10.1 *Precision*—The single-operator standard deviation for percentage of calcium carbonate from a single laboratory has been determined to be 0.24 % based on two sources with average calcium carbonate contents of 94.2 % and 96.7 %.

NOTE A1.5—Six samples were used from each of the two sources, and three replicate determinations were made on each sample. An inter laboratory study of this test method is being conducted to provide a complete precision statement.

A1.10.2 *Bias*—There is no accepted reference material suitable for determining the bias in this test method, therefore, no statement on bias is made.

A1.11. Keywords

A1.11.1 calcium carbonate; EDTA; limestone; magnesium carbonate; titration

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