



Standard Specification for Blended Hydraulic Cements¹

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This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This specification pertains to blended hydraulic cements for both general and special applications, using slag, pozzolan, limestone, or some combination of these, with portland cement or portland cement clinker or slag with lime.

NOTE 1—This specification prescribes ingredients and proportions, with some performance requirements whereas Performance Specification C1157 is a hydraulic cement specification in which performance criteria alone govern the products and their acceptance.

1.2 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. Values in SI units [or inch-pound units] shall be obtained by measurement in SI units [or inch-pound units] or by appropriate conversion, using the Rules for Conversion and Rounding given in [IEEE/ASTM SI 10](#), of measurements made in other units [or SI units]. Values are stated in only SI units when inch-pound units are not used in practice.

1.3 The text of this standard refers to notes and footnotes, which provide explanatory material. These notes and footnotes (excluding those in tables and figures) are not requirements of the standard.

2. Referenced Documents

2.1 ASTM Standards:²

- C51 Terminology Relating to Lime and Limestone (as used by the Industry)
- C109/C109M Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)

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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.

- C114 Test Methods for Chemical Analysis of Hydraulic Cement
- C150 Specification for Portland Cement
- C151 Test Method for Autoclave Expansion of Hydraulic Cement
- C157/C157M Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete
- C183 Practice for Sampling and the Amount of Testing of Hydraulic Cement
- C185 Test Method for Air Content of Hydraulic Cement Mortar
- C186 Test Method for Heat of Hydration of Hydraulic Cement
- C187 Test Method for Amount of Water Required for Normal Consistency of Hydraulic Cement Paste
- C188 Test Method for Density of Hydraulic Cement
- C191 Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle
- C204 Test Methods for Fineness of Hydraulic Cement by Air-Permeability Apparatus
- C219 Terminology Relating to Hydraulic Cement
- C226 Specification for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Hydraulic Cement
- C227 Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method)
- C311 Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete
- C430 Test Method for Fineness of Hydraulic Cement by the 45-μm (No. 325) Sieve
- C465 Specification for Processing Additions for Use in the Manufacture of Hydraulic Cements
- C511 Specification for Mixing Rooms, Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes
- C563 Test Method for Approximation of Optimum SO₃ in Hydraulic Cement Using Compressive Strength
- C688 Specification for Functional Additions for Use in Hydraulic Cements
- C821 Specification for Lime for Use with Pozzolans
- C1012 Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution
- C1038 Test Method for Expansion of Hydraulic Cement Mortar Bars Stored in Water



C1157 Performance Specification for Hydraulic Cement
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
E350 Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron
E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques
IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): the Modern Metric System

2.2 CSA Standards:

CSA A3004-D2 Determination of Total Organic Carbon in Limestone

3. Terminology

3.1 *Definitions*—The terms used in this specification are defined in Terminology **C219**, except for the following terms:

3.1.1 *binary blended cement, n*—a blended hydraulic cement consisting of portland cement with either a slag, a pozzolan, or a limestone.

3.1.2 *slag, n*—the term slag is used within this standard to denote either slag cement or granulated blast-furnace slag.

3.1.3 *ternary blended cement, n*—a blended hydraulic cement consisting of portland cement with either a combination of two different pozzolans, slag and a pozzolan, a pozzolan and a limestone, or a slag and a limestone.

NOTE 2—Relevant terms in Terminology **C219** applicable to this standard include portland cement, portland-cement clinker, hydraulic cement, slag cement, granulated blast-furnace slag, pozzolan, and calcium sulfate. Limestone is defined in Terminology **C51**.

4. Classification

4.1 This specification applies to the following types of blended cement that generally are intended for use as indicated.

4.1.1 Blended hydraulic cements for general concrete construction.

4.1.1.1 *Type IS*—Portland blast-furnace slag cement.

4.1.1.2 *Type IP*—Portland-pozzolan cement.

4.1.1.3 *Type IL*—Portland-limestone cement.

4.1.1.4 *Type IT*—Ternary blended cement.

4.2 *Reporting*:

4.2.1 The naming practice for blended cements shall be made by adding the suffix (X) to the type designation under **4.1.1**, where (X) equals the targeted percentage of slag, pozzolan or limestone, in the product expressed as a whole number by mass of the final blended product, within the allowable variation as stated in **15.3**.

4.2.2 The naming practice for ternary blended cements shall be made by adding the suffixes (AX) and (BY) to the Type IT designation under **4.1.1**, where:

A is either “S” for slag, “P” for pozzolan, or “L” for limestone,

whichever is present in larger amount by mass, and

X is the targeted percentage by mass of constituent A, and

B is either “S” for slag, “P” for pozzolan, or

“L” for limestone, and

Y is the targeted percentage by mass of constituent B.

Both X and Y values are expressed as a whole number by mass of the final blended product, within the allowable variation as stated in **15.3**. If X and Y are the same, list the two constituents in alphabetical order by constituent type (limestone, pozzolan, or slag).

NOTE 3—Examples of the naming practice in accordance with **4.2.1** and **4.2.2** are shown below (all percentages by mass):

Binary blended cement with 80 % portland cement and 20 % slag = Type IS(20).

Binary blended cement with 85 % portland cement and 15 % pozzolan = Type IP(15).

Binary blended cement with 90 % portland cement and 10 % limestone = Type IL(10).

Ternary blended cement with 70 % portland cement, 20 % slag and 10 % pozzolan = Type IT(S20)(P10).

Ternary blended cement with 65 % portland cement, 25 % of one pozzolan and 10 % of another pozzolan = Type IT(P25)(P10).

Ternary blended cement with 60 % portland cement and 20 % of slag and 20 % pozzolan = Type IT(P20)(S20).

Ternary blended cement with 80 % portland cement, 10 % limestone and 10 % pozzolan = Type IT(L10)(P10).

Ternary blended cement with 75 % portland cement, 15 % slag and 10 % limestone = Type IT(S15)(L10).

4.2.3 A simplified naming practice is used in this standard for practicality and clarity when referring to specific requirements for binary and ternary blended cements that are applicable to a range of products or in ternary blended cements when requirements are applicable to only one constituent within a specific range (%). (See **Note 4**.)

TABLE 1 Chemical Requirements

Cement Type ^A	Applicable Test Method	IS(<70), IT(P<S<70), IT(L<S<70)	IS(≥70), IT(S≥70)	IP, IT(P≥S), IT(P≥L)	IL, IT(L≥S), IT(L≥P)
Magnesium oxide (MgO), max, %	C114	...	...	6.0	...
Sulfate reported as SO ₃ , max, % ^B	C114	3.0	4.0	4.0	3.0
Sulfide reported as S ²⁻ , max, %	C114	2.0	2.0	...	...
Insoluble residue, max, % ^C	C114	1.0	1.0	...	...
Loss on ignition, max, %	C114	3.0 ^D	4.0 ^D	5.0 ^D	10.0

^A The chemical requirements in this table are applicable to all air-entrained cement equivalents.

^B It is permissible to exceed the values in the table for SO₃ content, provided it has been demonstrated by Test Method **C1038** that the cement with the increased SO₃ will not develop expansion exceeding 0.020 % at 14 days. When the manufacturer supplies cement under this provision, supporting data shall be supplied to the purchaser. See **Note 9**.

^C Insoluble residue maximum limit does not apply to ternary blended cements.

^D For ternary blended cements with limestone, loss on ignition is a maximum of 10.0 % by mass.



NOTE 4—Examples of the simplified naming practices in accordance with 4.2.3 are shown below:

1) An example when requirements are applicable to a range of products can be found in Table 1, where the maximum SO_3 content of 3 % applies to: binary blended cements with slag contents <70 %, indicated as IS(<70); and ternary blended cements with a pozzolan content less than the slag content and the slag content is less than 70 %, indicated as IT($P < S < 70$).

2) An example when requirements are applicable to only one constituent within a specific range (%) of that constituent can be found in 9.2, where testing is required only when the slag content is <25 %. Because the requirement is based on the slag content only with no relation to the pozzolan or limestone content, a simplified naming practice is employed and the range of ternary blended cements is indicated as Type IT($S < 25$).

4.3 Special Properties:

4.3.1 Air-entraining cement, when desired by the purchaser, shall be specified by adding the suffix (A) to the type designation under 4.1.1.

NOTE 5—A given mass of blended cement has a larger absolute volume than the same mass of portland cement. This should be taken into consideration in purchasing cements and in proportioning concrete mixtures.

4.3.2 Moderate heat of hydration, when desired by the purchaser, shall be specified by adding the suffix (MH) to the type designation under 4.1.1.

4.3.3 Moderate sulfate resistance, when desired by the purchaser, shall be specified by adding the suffix (MS) to the type designation under 4.1.1.

4.3.4 High sulfate resistance, when desired by the purchaser, shall be specified by adding the suffix (HS) to the type designation under 4.1.1.

4.3.5 Low heat of hydration, when desired by the purchaser, shall be specified by adding the suffix (LH) to the type designation under 4.1.1.

4.3.6 Resistance to alkali-silica reactive aggregate expansion, when desired by the purchaser, shall be specified by adding the suffix (R) to the type designation under 4.1.1.

NOTE 6—Special characteristics attributable to slag, pozzolan or limestone will vary based on quantities contained within the blended cements. Type IT cements with greater than 5 % limestone and Type IL cements are not permitted as moderate (MS) or high (HS) sulfate resistance cements, pending results of further research.

5. Ordering Information

5.1 Orders for material under this specification shall include the following:

- 5.1.1 Specification number,
- 5.1.2 Type or types required,
 - 5.1.2.1 Indicate allowable slag, pozzolan, or limestone maximum or minimum percentage by mass, if required.
- 5.1.3 Optional special properties required (see 4.3):
 - 5.1.3.1 MS if moderate sulfate resistance is required;
 - 5.1.3.2 HS if high sulfate resistance is required;
 - 5.1.3.3 MH if moderate heat of hydration is required;
 - 5.1.3.4 LH if low heat of hydration is required;
 - 5.1.3.5 R if resistance to alkali-silica reactive aggregate expansion is required;
 - 5.1.3.6 A if air entraining is required;
 - 5.1.3.7 Accelerating addition, if required;
 - 5.1.3.8 Retarding addition, if required;
 - 5.1.3.9 Water reducing addition, if required;

5.1.3.10 Water reducing and accelerating addition, if required; and

5.1.3.11 Water reducing and retarding addition, if required.

5.1.4 Certification, if desired (see Section 15).

NOTE 7—It is important to check for availability of various options. Some multiple options are mutually incompatible or unattainable.

6. Materials

6.1 Materials permitted to be used as ingredients in cements manufactured to comply with this specification are:

6.1.1 *Portland Cement*—For purposes of this specification, portland cement meeting the requirements of Specification C150 is suitable. Portland cement or other hydraulic materials, or both, containing high free lime are not prohibited from use as long as the autoclave test limits for the blended cement are met.

6.1.2 *Portland Cement Clinker*.

6.1.3 *Slag*—Slag shall be slag cement or granulated blast-furnace slag and comply with requirements in Section 9.

6.1.4 *Pozzolan*—Pozzolan shall comply with requirements in Section 9.

6.1.5 *Limestone*—Limestone, as defined in Terminology C51, shall be naturally occurring and comply with the requirements indicated in 8.2.

6.1.6 *Hydrated Lime*—Hydrated lime used as part of a blended cement shall meet the requirements of Specification C821, except that when interground in the production process there shall be no minimum fineness requirement.

6.1.7 *Air-Entraining Addition*—When air-entraining cement is specified, an addition meeting the requirements of Specification C226 shall be used.

6.1.8 When processing additions are used in the manufacture of cement, they shall have been shown to meet the requirements of Specification C465 in the amounts used or greater (see subsection 15.2).

6.1.9 When functional additions (used at the sole option of the purchaser) are used they shall have been shown to meet the requirements of Specification C688 when tested with the cement to be used, in the amount used or greater (see subsection 15.2).

6.1.10 *Other Additions*—The cement covered by this specification shall contain no additions except as provided for above except that water or calcium sulfate (see Terminology C219), or both, if added, shall be in amounts so that the limits shown in Table 1 for sulfate reported as SO_3 and loss on ignition are not exceeded.

7. Manufacture

7.1 *Binary Blended Cement*—Binary blended cement shall be a hydraulic cement consisting of an intimate and uniform blend (see Note 8) produced either by intergrinding or blending or by a combination of intergrinding and blending portland cement clinker or portland cement with a pozzolan or a slag, or a limestone. Any slag, pozzolan, or limestone used as an ingredient or addition in portland cement used to manufacture a binary blended cement shall be included in the total amount of those materials reported in 4.2 or 15.1. The following requirements shall apply:



7.1.1 Portland Blast-Furnace Slag Cement—Portland blast-furnace slag cement shall be a hydraulic cement in which the slag constituent is up to 95 % by mass of the blended cement. Portland blast-furnace slag cement with a slag content equal to or exceeding 70 % by mass, is permitted to contain hydrated lime.

7.1.2 Air-Entraining Portland Blast-Furnace Slag Cement—Air-entraining portland blast-furnace slag cement shall be portland blast-furnace slag cement to which sufficient air-entraining addition has been added so that the resulting product complies with the air content of mortar requirements.

7.1.3 Portland-Pozzolan Cement—Portland-pozzolan cement shall be a hydraulic cement in which the pozzolan constituent is up to 40 % by mass of the blended cement.

7.1.4 Air-Entraining Portland-Pozzolan Cement—Air-entraining portland-pozzolan cement shall be portland-pozzolan cement to which sufficient air-entraining addition has been added so that the resulting product complies with the air content of mortar requirements.

7.1.5 Portland-Limestone Cement—Portland-limestone cement shall be a hydraulic cement in which the limestone content is more than 5 % but less than or equal to 15 % by mass of the blended cement.

7.1.6 Air-Entraining Portland-Limestone Cement—Air-entraining portland-limestone cement shall be portland-limestone cement to which sufficient air-entraining addition has been added so that the resulting product complies with the air content of mortar requirements.

7.2 Ternary Blended Cement—Ternary blended cement shall be a hydraulic cement consisting of an intimate and uniform blend (see **Note 8**) produced either by intergrinding, by blending, or a combination of intergrinding, and blending portland cement clinker or portland cement with (1) two different pozzolans, (2) slag and a pozzolan, (3) a pozzolan and a limestone, or (4) a slag and a limestone. Ternary blended cement Type IT(S $\geq$ 70) shall have a maximum limestone content of 15 % by mass and is permitted to contain hydrated lime. All other ternary blended cements shall have a maximum pozzolan content of 40 % by mass of the blended cement, a maximum limestone content of 15 % by mass of the blended cement, and the total content of pozzolan, limestone, and slag shall be less than 70 % by mass of the blended cement. Any slag, pozzolan, or limestone used as ingredient in portland cement used to manufacture a blended cement shall be included in the total amount of those materials reported in **4.2** or **15.1**.

NOTE 8—The attainment of an intimate and uniform blend of two or more types of fine materials is difficult. Consequently, adequate equipment and controls must be provided by the manufacturer. The purchasers should assure themselves of the adequacy of the blending operation.

8. Chemical Composition

8.1 Blended Cement—Cement of the type specified shall conform to the applicable chemical requirements prescribed in **Table 1**.

NOTE 9—There are cases where performance of a cement is improved with SO₃ in excess of the **Table 1** limits in this specification. Test Method **C563** is one of several methods a manufacturer can use to evaluate the effect of sulfate content on cement characteristics. Whenever SO₃ content

of a cement exceeds **Table 1** limits, Test Method **C1038** results provide evidence that excessive expansion does not occur at this higher sulfate content.

8.1.1 If the purchaser has requested the manufacturer to state in writing the composition of the blended cement purchased, the composition of the cement furnished shall conform to that shown in the statement within the following tolerances (see **Note 10**).

	Tolerance, $\pm$ %
Silicon dioxide (SiO ₂)	3
Aluminum oxide (Al ₂ O ₃)	2
Calcium oxide (CaO)	3

NOTE 10—This means that if the manufacturer's statement of the composition says "SiO₂: 32 %," the cement when analyzed, shall be found to contain between 29 and 35 % SiO₂.

8.2 Limestone—Limestone for use in the manufacture of portland-limestone cement, or a ternary blended cement in which limestone is an ingredient, shall have a calcium carbonate content of at least 70 % by mass. Such limestone shall meet the requirements of **Table 2** for methylene blue index and total organic carbon content.

8.3 Pozzolan—Pozzolan for use in the manufacture of portland-pozzolan cement, or a ternary blended cement in which pozzolan is an ingredient, shall meet the loss-on-ignition requirement of **Table 4**.

9. Physical Properties

9.1 Blended Cement—Blended cement of the type specified shall conform to the applicable physical requirements prescribed in **Table 3**. When specified, blended cement with special properties shall conform to applicable physical requirements in **Table 4**, and requirements for compressive strength and air content in **Table 4** supersede those in **Table 3**.

9.2 Pozzolan or Slag—Pozzolan or slag that is to be blended with cement shall be tested in the same state of subdivision as that in which it is to be blended. Pozzolan shall conform to the fineness requirement and the activity index requirement of **Table 5**. Slag that is to be used for portland blast-furnace slag cements Type IS(<25) or ternary blended cements Type IT(S<25) shall conform to the activity index requirement of **Table 5**. Such pozzolan, or slag that is to be interground with portland cement clinker shall, before testing for conformance with requirements of **Table 5**, be ground in the laboratory to a fineness at which it is believed to be present in the finished cement. It is the manufacturer's responsibility to decide on the fineness at which the testing is to be carried out, and when

TABLE 2 Requirements for Limestone for Use in Blended Cements

	Applicable Test Method	
CaCO ₃ content, min. % by mass	C114	70 ^A
Methylene blue index, max, g/100 g	See Annex A2	1.2
Total organic carbon, max, % by mass	See Annex A3	0.5

^A The calcium carbonate content of limestone shall be determined by multiplying the CaO content of the limestone determined by Test Methods **C114** by a factor of 1.785.



TABLE 3 Physical Requirements for Blended Cements

Cement Type	Applicable Test Method	IL, IP, IS(<70), IT(S<70)	IS(≥70), IT(S≥70)
Fineness	C204, C430	A	A
Autoclave expansion, max, % ^B	C151	0.80	0.80
Autoclave contraction, max, % ^B	C151	0.20	0.20
Time of initial setting, Vicat test: ^C			
Set, minutes, not less than	C191	45	45
Set, hours, not more than		7	7
Air content of mortar, volume %, max	C185	12	12
Compressive strength, min, MPa [psi]:			
3 days	C109/C109M	13.0 [1890]	...
7 days		20.0 [2900]	5.0 [720]
28 days		25.0 [3620]	11.0 [1600]

^A Both amount retained when wet-sieved on 45-μm (No. 325) sieve and specific surface by air permeability apparatus, m²/kg, shall be reported on all mill test reports requested under 15.4.

^B The specimens shall remain firm and hard and show no signs of distortion, cracking, checking, pitting, or disintegration when subjected to the autoclave expansion test.

^C The time of setting of cements containing a user-requested accelerating or retarding functional addition need not meet the limits of this table, but shall be stated by the manufacturer.

TABLE 4 Physical Requirements for Blended Cements with Special Properties

Special Property Designation ^A	Applicable Test Method	A	MS ^B	HS ^B	MH	LH	R ^C
Air content of mortar:							
minimum, volume %	C185	16 ^D	...	...	...	...	...
maximum, volume %		22 ^D	12	12	12	12	12
Compressive strength, ^E min, MPa [psi]:							
3 days	C109/C109M	10.4 [1510]	11.0 [1600]	11.0 [1600]	10.4 [1510]	...	13.0 [1890]
7 days		16.0 [2320]	18.0 [2610]	18.0 [2610]	16.0 [2320]	11.0 [1600]	20.0 [2900]
28 days		20.0 [2900]	25.0 [3620]	25.0 [3620]	20.0 [2900]	21.0 [3050]	25.0 [3620]
Heat of hydration, max, kJ/kg [cal/g]:							
7 days	C186	...	...	...	290 [70]	250 [60]	...
28 days		...	...	...	330 [80]	290 [70]	...
Water requirement, max weight % of cement	C109/C109M	...	...	...	...	64	...
Drying shrinkage, max, %	C157/C157M	...	...	...	...	0.15	...
Sulfate resistance, max, %:							
Expansion at 180 days	C1012	...	0.10	0.05 ^F	...	...	...
Expansion at 1 year		...	...	0.10 ^F	...	...	...
Mortar expansion, max, %:							
14 days	C227	...	...	...	...	...	0.020
8 weeks		...	...	...	...	...	0.060

^A These requirements apply only if specified and are designated by suffixes A, MS, HS, MH, LH, or R as appropriate to type designations IL, IP, IS(<70), or IT(S<70). See 4.3. Requirements for fineness, autoclave expansion, autoclave contraction, and time of setting shall conform to Table 3.

^B Type IT cements with greater than 5% limestone and Type IL cements are not permitted as moderate (MS) or high (HS) sulfate resistance cements.

^C Compliance with this requirement shall not be required unless the cement will be used with alkali-silica reactive aggregate.

^D These air content requirements apply to cements with multiple special property designations when one of those designations is (A).

^E When multiple special property designations are applied, the set of strength requirements for the special property designation with the lowest 7-day minimum strength requirement shall apply.

^F Testing at one year shall not be required when the cement meets the 180-day limit. A cement failing the 180-day limit shall not be rejected unless it also fails the one-year limit.

requested to do so by a purchaser, to report the information upon which the decision was based.

9.3 Pozzolan for use in the manufacture of portland-pozzolan cement, Type IP(<15) and IP(<15)-A or ternary blended cements Type IT(P<15) and Type IT(P<15)-A, shall meet the requirements of Table 5 when tested for mortar expansion of pozzolan as described in 11.1.13. If the alkali content of the clinker to be used for the production lots changes by more than 0.2 % total as equivalent Na₂O, calculated as Na₂O + 0.658 K₂O, from that of the clinker with which the acceptance tests were carried out, the pozzolan shall be retested to show compliance with the requirements of Table 5.

10. Sampling

10.1 Sample the materials in accordance with the following methods:

10.1.1 *Sampling Blended Cements*—Practice C183.

10.1.1.1 When the purchaser desires that the cement be sampled and tested to verify compliance with this specification, perform sampling and testing in accordance with Practice C183.

10.1.1.2 Practice C183 is not designed for manufacturing quality control and is not required for manufacturer's certification.

**TABLE 5 Requirements for Pozzolan for Use in Blended Cements and for Slag for Use in Portland Blast-Furnace Slag Cement Type IS(<25) and Ternary Blended Cement Type IT(S<25)**

Pozzolan and Slag, as applicable	Applicable Test Method	
Fineness; Amount retained when wet-sieved on 45- μ m (No. 325) sieve, max, %	C430	20.0
Alkali reactivity of pozzolan for use in Types IP(<15); IT(P<15) and IP(<15)-A; IT(P<15)-A cements, six tests, mortar bar expansion at 91 days, max, %	C227	0.05
Activity index with portland cement, at 28 days, min, %	(see Annex A1)	75
Loss on ignition of pozzolan, max, %	C311	10.0
Natural pozzolan		6.0
Fly ash		6.0
Silica fume		6.0

TABLE 6 Aggregate Grading Requirements for Mortar Expansion Test

Sieve Size		Weight %
Passing	Retained on	
4.75-mm (No. 4)	2.36-mm (No. 8)	10
2.36-mm (No. 8)	1.18-mm (No. 16)	25
1.18-mm (No. 16)	600- μ m (No. 30)	25
600- μ m (No. 30)	300- μ m (No. 50)	25
300- μ m (No. 50)	150- μ m (No. 100)	15

10.1.2 *Sampling Pozzolan*—Test Methods C311. One 2 kg [4 lb] sample shall be taken from approximately each 360 Mg [400 tons] of pozzolan.

11. Test Methods

11.1 Determine the applicable properties enumerated in this specification in accordance with the following test methods:

11.1.1 *Chemical Analysis*—Test Methods C114, with the special provisions noted therein applicable to blended cement analyses.

11.1.2 *Fineness by Sieving*—Test Method C430.

11.1.3 *Fineness by Air-Permeability Apparatus*—Test Method C204.

11.1.4 *Autoclave Expansion*—Test Method C151, except that, in the case of portland blast-furnace slag cement Type IS(≥ 70) or ternary blended cement Type IT(S ≥ 70), the test specimens shall remain in the moist cabinet for a period of 48 h before being measured for length, and the neat cement shall be mixed for not less than 3 min nor more than 3½ min.

11.1.5 *Time of Setting*—Test Method C191.

11.1.6 *Air Content of Mortar*—Test Method C185, using the actual specific gravity of the cement, if it differs from 3.15 by more than 0.05, in calculating the air content.

11.1.7 *Compressive Strength*—Test Method C109/C109M.

11.1.8 *Heat of Hydration*—Test Method C186.

11.1.9 *Normal Consistency*—Test Method C187, except that in the case of portland blast-furnace slag cement IS(≥ 70) or ternary blended cement IT(S ≥ 70), the paste shall be mixed for not less than 3 min nor more than 3½ min.

11.1.10 *Specific Gravity*—Test Method C188.

11.1.11 *Water Requirement*—The mass of mixing water added to the six-cube batch in accordance with Test Method C109/C109M, as a percentage of the total cementing ingredients.

11.1.12 *Mortar Expansion of Blended Cement*—Test Method C227, using crushed Pyrex glass No. 7740³ as aggregate and the grading prescribed in Table 6.

11.1.13 *Mortar Expansion of Pozzolan for Use in Portland-Pozzolan Cement Types IP(<15) and IP(<15)-A or Ternary Blended Cement Types IT(P<15) and IT(P<15)-A*—Using the pozzolan and the clinker or cement that are to be used together in the production of the blended cement, prepare portland-pozzolan cements Types IP(<15) and IP(<15)-A or ternary blended cement Types IT(P<15) and IT(P<15)-A containing 2.5, 5, 7.5, 10, 12.5, and 15 mass % of the pozzolan. These blends shall be tested in accordance with Test Method C227 using a sand judged to be a nonreactive by the mortar bar test in Test Method C227. The expansion of the mortar bars shall be measured at 91 days, and all the six blends shall meet the expansion requirement in Table 5.

11.1.14 *Drying Shrinkage*—Test Method C157/C157M. Make three specimens using the proportion of dry materials of 1 part of cement to 2.75 parts of Test Method C109/C109M graded Ottawa sand. Use a curing period of 6 days and an air storage period of 28 days. Report the linear contraction during air storage based on an initial measurement after the 6-day water-curing period.

11.1.15 *Activity Index with Portland Cement*—Test in accordance with Annex A1.

11.1.16 *Sulfate Resistance*—see Test Method C1012

11.1.17 *Methylene Blue Index of Limestone*—Annex A2.

11.1.18 *Total Organic Carbon Content of Limestone*—Annex A3.

11.1.19 *Loss on Ignition of Pozzolan*—Test Methods C311.

12. Testing Time Requirements

12.1 The following periods from time of sampling shall be allowed for the completion of testing:

3-day test	8 days
7-day test	12 days
14-day test	19 days
28-day test	33 days
8-week test	61 days

13. Inspection

13.1 Facilities shall be provided to the purchaser for careful inspection and sampling of the finished cement. Inspection and sampling of finished cement shall be at the mill or distribution site controlled by the manufacturer, or at any other location as agreed by the purchaser and seller.

13.2 The manufacturer shall provide suitable facilities to enable the inspector to check the relative masses of the constituents used, and the intergrinding or blending operation

³ Pyrex Glass No. 7740 is available as lump cullet from the Corning Glass Works, Corning, NY; this is the sole source of supply of the apparatus known to the committee at this time. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

used to produce the cement. The plant facilities for intergrinding or blending and inspection shall be adequate to ensure compliance with the provisions of this specification.

14. Rejection

14.1 At the option of the purchaser, cement shall be rejected if it fails to meet any of the requirements of this specification applicable to the cement. Such rejection shall apply to an optional requirement only if that option has been invoked for the cement.

14.2 When the purchaser requires, cement in bulk storage for a period greater than six months shall be resampled and retested and, at the option of the purchaser, shall be rejected if it fails to meet any of the applicable requirements of this specification. Cement so rejected shall be the responsibility of the owner of record at the time of sampling for retest.

14.3 When the purchaser requires, packages more than 2 % below the mass marked thereon shall be rejected; or if the average mass of packages in any shipment, as shown by determining the mass of 50 packages taken at random, is less than that marked on the packages, the entire shipment, at the option of the purchaser, shall be rejected.

15. Certification

15.1 At the request of the purchaser, the manufacturer shall state in writing the source, amount, and composition of the essential constituents used in manufacture of the finished cement and the composition of the blended cement purchased.

15.2 At the request of the purchaser, the manufacturer shall state in writing the nature, amount, and identity of any processing, functional, or air-entraining addition used; and also, if requested, shall supply test data showing compliance of any such processing addition with the provisions of Specification C465 and of any such functional addition with the provisions of Specification C688, and of any such air-entraining addition with the provisions of Specification C226.

15.3 At the request of the purchaser, the manufacturer shall also state in writing that the amounts of pozzolan, slag, or

limestone in the finished cement will not vary more than ± 5.0 mass % of the finished cement from lot to lot or within a lot.

15.4 Upon request of the purchaser in the contract or order, a manufacturer's certification shall be furnished indicating that the material was tested during production or transfer in accordance with this specification, that it complies with this specification, and a report of the test results shall be furnished at the time of shipment (to include both amount retained on the 45- μ m (No. 325) sieve and specific surface by the air permeability method).

16. Packaging and Package Marking

16.1 When the cement is delivered in packages, the words, "portland blast-furnace slag cement," "portland-pozzolan cement," "portland-limestone cement," or "ternary blended cement" as appropriate; the type of cement, name and brand of the manufacturer, and the mass of the cement contained therein, shall be plainly marked on each package. When the cement contains a functional addition listed in 5.1.3.6 – 5.1.3.11, the type of functional addition shall be plainly marked on each package. Similar information shall be provided in the shipping documents accompanying the shipment of packaged or bulk cement. All packages shall be in good condition at the time of inspection.

17. Storage

17.1 The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment, and in a suitable weathertight building that will protect the cement from dampness and minimize warehouse set.

18. Keywords

18.1 blended hydraulic cement; fly ash cement; hydraulic cement; portland blast-furnace slag cement; portland-limestone cement; portland-pozzolan cement; pozzolanic cement; slag; granulated blast-furnace slag

ANNEXES

(Mandatory Information)

A1. ACTIVITY INDEX WITH PORTLAND CEMENT

A1.1. Specimen Preparation

A1.1.1 Mold, cure, and test the specimens from a control mix and from a test mix in accordance with Test Method C109/C109M. The portland cement used in the control mix shall meet the requirements of Specification C150, and shall be the type, and if available, the brand of cement to be used in the work. Make three-cube batches as follows: (For 6- or 9-cube batches, double or triple, respectively, the amounts of dry ingredients.)

A1.1.1.1 Control Mix:

250 g of portland cement
687.5 g of graded Ottawa sand
X mL of water required for flow of 100 to 115

A1.1.1.2 Pozzolan Test Mix:

162.5 g of portland cement
g of pozzolan:
87.5 $\times$ sp gr of the sample/sp gr of the portland cement
687.5 g of graded Ottawa sand



Y mL of water required for flow of 100 to 115

A1.1.1.3 Slag Test Mix:

75 g of portland cement

g of slag:

175 × sp gr of the slag/sp gr of the portland cement

687.5 g of graded Ottawa sand

Z mL of water required for flow of 100 to 115

A1.2 Storage of Specimens

A1.2.1 After molding, place the specimens and molds (on the base plates) in the moist room or closet at $23.0 \pm 2.0^{\circ}\text{C}$ [$73.5 \pm 3.5^{\circ}\text{F}$] for 20 to 24 h. While in the moist room or closet, protect the surface from dripping water. Remove the molds from the moist room or closet and remove the cubes from the molds. Place the cubes in close-fitting metal or glass containers (**Note A1.1**), seal the containers airtight, and store at $38.0 \pm 2.0^{\circ}\text{C}$ [$100.0 \pm 3.5^{\circ}\text{F}$] for 27 days. Allow the specimens to cool to $23.0 \pm 2.0^{\circ}\text{C}$ [$73.5 \pm 3.5^{\circ}\text{F}$] before testing.

NOTE A1.1—Use any metal container having a capacity of three cubes if it can be sealed airtight by soldering. Containers of light-tinned sheet metal with inside dimensions of 52 by 52 by 160 mm [2 by 2 by 6.25 in.] have been found to be satisfactory. Wide-mouth Mason jars of 1-L capacity have been found to be satisfactory, provided care is taken to prevent breakage.

(Warning)—Fresh hydraulic cementitious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure.)⁴

⁴ Section on Safety, Manual of Cement Testing, *Annual Book of ASTM Standards*, Vol 04.01.

A1.3 Compressive Strength Test

A1.3.1 Determine the compressive strength of the three specimens of the control mix and of the test mix at an age of 28 days in accordance with Test Method **C109/C109M**.

A1.4. Calculation

A1.4.1 Calculate the activity index with portland cement as follows:

$$\text{Activity index with portland cement} = (A/B) \times 100 \quad (\text{A1.1})$$

where:

A = average compressive strength of test mix cubes, MPa, and

B = average compressive strength of control mix cubes, MPa.

A1.5. Precision and Bias

A1.5.1 *Precision*—Single operator precision, on blended cements using fly ash is essentially the same as on fly ash/cement blends in Research Report RR:C09-1001⁵ and it was found to have 3.8 % coefficient of variation (1s %). This indicates that results of two properly conducted tests by the same operator are not expected to differ by more than 10.7 % (d2s) of the average of two results. Since the test is performed solely for the purpose of manufacturer certification of raw material quality, no multilaboratory precision is applicable.

A1.5.2 *Bias*—Since there are no standard reference materials, bias cannot be determined.

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C09-1001. Contact ASTM Customer Service at service@astm.org.

A2. METHYLENE BLUE INDEX TEST FOR LIMESTONE

A2.1. Scope

A2.1.1 This annex describes the laboratory procedures for the quantitative determination of methylene blue dye adsorption index of limestone for use as an ingredient in blended cement.

A2.1.2 The text of this annex references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the annex.

A2.1.3 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this annex.

A2.1.4 *This annex 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.*

A2.2. Summary of Test

A2.2.1 The sample is reduced to a fine powder prior to testing. Increments of methylene blue dye solution are added successively to a suspension of the prepared test portion in water. The adsorption of dye solution by the suspension is checked after each addition of solution by carrying out a stain test on filter paper to detect the presence of free dye. When the presence of free dye is confirmed, the methylene blue index value (*MBI*) is calculated and expressed as grams of dye adsorbed per 100 g of the sample tested.

A2.3. Significance and Use

A2.3.1 This annex provides a means to determine the amount of methylene blue dye adsorbed by a sample of finely ground limestone. Methylene blue dye is preferentially adsorbed by clay minerals that may be present in limestone. The amount of dye adsorbed is related to the clay type and content. Certain clays may increase the water demand or affect other

properties of cements if present in sufficient quantity in the limestone when used as an ingredient in cement. The specification places a limit on the methylene blue index of limestone permitted as an ingredient in blended cement.

A2.4. Apparatus

A2.4.1 The equipment and materials, including the temperature and humidity of the testing room, dry materials, and mixing water, shall meet the requirements of Specification C511, unless otherwise specified.

A2.4.2 The following equipment shall be included to perform this test:

- (a) burette, with capacity of either 100 mL or 50 mL and graduation of either $\frac{1}{10}$ mL or $\frac{1}{5}$ mL, or one 5 mL and one 2 mL micro-pipette;
- (b) filter paper, quantitative and ash-free ($<0.010\%$); 95 g/m^2 ; thickness 0.20 mm; filtration speed 75 s; pore size 20 μm ;
- (c) glass rod, 300 mm in length and 8 mm in diameter;
- (d) impeller agitator, capable of controlled variable rotation rates up to 600 ± 60 r/min with either three or four impeller blades of 75 ± 10 mm diameter (see Note A2.1);
- (e) balance, readable to 0.1 % of the mass to be weighed;
- (f) stopwatch or stopclock, readable to 1 s;
- (g) test sieve, 2 mm aperture, with guard sieve, if applicable;
- (h) beaker, glass or plastic, capacity about 1 L or about 2 L;
- (i) volumetric flask, capacity 1 L;
- (j) ventilated oven, thermostatically controlled to maintain a temperature of $105 \pm 5^\circ\text{C}$ (see Note A2.2);
- (k) thermometer, readable to 1°C ;
- (l) spatula;
- (m) desiccator;
- (n) tinted-glass storage bottle; and
- (o) a 63- μm (No. 230) sieve conforming to the requirements of Specification E11.

NOTE A2.1—Alternative types of mixers can be used if it can be shown that results obtained agree with results produced using an impeller agitator as described in A2.4.2(d).

A2.5. Reagents and Materials

A2.5.1 The following materials shall be included to perform this test:

- (a) methylene blue ($\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S}\cdot n\text{H}_2\text{O}$ ($n = 2$ to 3)) purity $\geq 98.5\%$;
- (b) kaolinite; and
- (c) water.

A2.6. Procedure

A2.6.1 Preparation of Methylene Blue Solution (10 g/L):

A2.6.1.1 *Determination of Moisture Content of Methylene Blue Powder*—The determination of the moisture content of methylene blue powder shall be as follows:

- (a) Weigh 5 ± 0.01 g of methylene blue powder and record the mass;
- (b) Dry the powder at $100 \pm 5^\circ\text{C}$ to constant mass (see Note A2.2);

(c) Cool the powder in the desiccator and weigh immediately after taking out of the desiccator, and record the mass to the nearest 0.01 g;

(d) Calculate and record the water content of the methylene blue, to the nearest 0.01 % by mass, using the following equation:

$$W = ((M_h - M_g)/M_h) \times 100 \quad (\text{A2.1})$$

where:

- W = moisture content, % by mass,
- M_h = mass of methylene blue powder, g, and
- M_g = mass of dried methylene blue powder, g.

The moisture content shall be determined for the preparation of every new batch of methylene blue dye solution.

NOTE A2.2—Above 105°C , methylene blue powder can be altered.

A2.6.1.2 *Preparation of Methylene Blue Dye Solution*—The preparation of methylene blue dye solution shall be as follows:

(a) Calculate the mass of methylene blue powder required to provide 10 ± 0.01 g of dry powder according to the equation:

$$M = (100 + W)/10 \quad (\text{A2.2})$$

where:

- M = mass of undried (as received) methylene blue powder, g, and
- W = moisture content of powder as determined in subsection A2.6.1.1, % by mass.

(b) Warm between 500 and 700 mL of distilled or demineralized water in a beaker not exceeding 40°C ;

(c) Agitate the contents of the beaker while slowly pouring the methylene blue powder as calculated in A2.6.1.2(a) into warm water;

(d) Continue to agitate for 45 min to achieve complete dissolution of the powder, and then allow to cool to 20°C ;

(e) Pour the solution quantitatively into a 1 L volumetric flask, ensuring the flask and water are at a temperature of $20 \pm 1^\circ\text{C}$, and dilute to volume;

(f) Shake the flask to ensure complete mixing and transfer the contents into a tinted-glass storage bottle;

(g) Mark the storage bottle with the preparation name, preparation date, expiration date and the solution strength “10 g/L methylene blue.” Methylene blue solution older than 28 days shall not be used.

A2.6.2 *Description of the Stain Test*—After each injection of dye, the stain test shall be conducted by taking a drop of suspension by means of a glass rod and depositing it on the filter paper. The stain which is formed is composed of a central deposit of material, of a generally solid blue color, surrounded by a colorless wet zone. The amount of drop taken shall be such that the diameter of the deposit is between 8 and 12 mm. The test shall be deemed positive if, in the wet zone, a halo consisting of a persistent light blue ring of about 1 mm is formed around the central deposit.

NOTE A2.3—As the end-point is approached, the halo will appear, but can then disappear again because the clay minerals can take some time to complete their adsorption of the dye. For this reason the end-point is confirmed by repeating the stain test at 1 min intervals for 5 min without adding more dye solution.

NOTE A2.4—A test on kaolinite of known MB_K value should be carried out at regular intervals to check the constancy of results. This procedure should also be used to check a new dye solution.

A2.6.3 Sample Preparation:

A2.6.3.1 Kaolinite—Dry the kaolinite to constant mass at $110 \pm 5^\circ\text{C}$ in a quantity sufficient to obtain at least 30 g of dry material. Allow the sample to cool in a desiccator to constant mass.

A2.6.3.2 Limestone—A sample of at least 100 g shall be dried at $110 \pm 5^\circ\text{C}$ to constant mass and allowed to cool in a desiccator. Reduce the dried sample to pass through a 63- μm (No. 230) sieve.

NOTE A2.5—Samples ground to completely pass through 212- μm and 75- μm (Specification E11 No. 70 and No. 200) sieves have been found to yield similar and consistent results on a limestone with a known (for example, previously determined) methylene blue value. The approximate Blaine fineness results on these sieves were found to be 250 m^2/kg and 630 m^2/kg , respectively. Experience in Europe suggests passing through a 63- μm sieve (Specification E11 No. 230) is adequate in approximating similar Blaine fineness.

A2.6.4 Suspension Preparation—Prepare the sample suspension as follows:

(a) If an impeller agitator is used, ensure the impeller of the mixer is approximately 10 mm above the base of the mixing container.

(b) Stir the dye solution or, alternatively, mix it thoroughly, fill the burette with dye solution, and return the stock of dye solution to a dark place.

(c) Weigh 30.0 ± 0.1 g of dry sample (kaolinite or limestone) and record the mass of kaolinite as M_{KI} , limestone mass as M_{LI} .

(d) Pour the sample into a beaker containing 500 mL of distilled or demineralized water, stirring at sufficient speed to disperse the sample as added.

(e) Increase the speed of agitation to ensure the contents are completely in suspension, and agitate the contents of the beaker for 5 minutes (see Note A2.6).

(f) Inject 5 mL of the methylene blue dye solution into the suspension and reduce the agitation speed to 400 ± 40 r/min for the remainder of the test.

(g) Carry out the stain test in accordance with A2.6.5.

(h) If insufficient fines are present in the limestone test portion to obtain a halo, 30.0 ± 0.1 g of kaolinite (dried at 110°C) should be added together with V_K mL of dye solution, where V_K = the volume of dye solution adsorbed by 30 g of kaolinite.

NOTE A2.6—For an impeller agitator, agitator rotation speeds up to 600 r/min $\pm$ 60 r/min are required.

A2.6.5 Determination of the Quantity of Dye Adsorbed—Determine the quantity of dye adsorbed as follows:

(a) Place the filter paper on top of an empty beaker, or some other suitable support, so that most of its surface is not in contact with any solid or liquid.

(b) After at least 1 min of agitating at the slower speed of 400 ± 40 r/min following the injection of the dye, carry out a stain test on the filter paper.

(c) Ensure that the amount of the drop taken is such that the deposit has a diameter of between 8 and 12 mm.

(d) If after the addition of the initial 5 mL of dye solution the halo does not appear, add a further 5 mL of dye solution, continue agitating for 1 min, and carry out another stain test.

(e) If a halo still does not appear, continue agitating, making additions of dye and doing stain tests in this manner until a halo is observed. When this stage is reached, continue agitating and without further additions of dye solution, perform stain tests at 1 min intervals.

(f) If the halo disappears during the first 4 min, add a further 5 mL of dye solution. If the halo disappears during the fifth minute, add only 2 mL of dye solution. In either case, continue agitating and doing stain tests until a halo persists for 5 min.

(g) Record the total volume of dye solution (for kaolinite as V_K , for limestone as V_T) added to produce a halo that persists for 5 min, to the nearest 1 mL.

(h) If for the limestone test portion a halo does not appear, repeat the test with the addition of materials as described in subsection A2.6.4(h).

(i) If the sample under test was kaolinite, calculate and record the MB_K value in accordance with Eq A2.3. For limestone, calculate and record the MB value in accordance with Eq A2.4.

NOTE A2.7—Containers should be cleansed thoroughly with water as soon as the tests are completed. Traces of any detergents used should be removed by thorough rinsing. It is recommended that containers used in methylene blue tests are reserved specifically for that test.

A2.7. Calculation and Reporting

A2.7.1 The methylene blue value of kaolinite, MB_K , expressed in g of dye per 100 g of the processed sample shall be given by the following equation:

$$MB_K = V_K / M_{KI} \quad (\text{A2.3})$$

where:

M_{KI} = the mass of the kaolinite sample, g, and

V_K = the total volume of dye solution injected, mL.

The MB_K value shall be recorded to the nearest 0.01 g of dye per 100 g of the processed sample.

A2.7.2 The methylene blue value of limestone, MB , expressed in g of dye per 100 g of the processed sample shall be given by the following equation:

$$MB = (V_T - V_K) / M_{LI} \quad (\text{A2.4})$$

where:

V_K = volume of dye solution adsorbed by 30 g kaolinite, if kaolinite and additional dye added, mL,

M_{LI} = the mass of the test portion, g, and

V_T = the total volume of dye solution injected, mL.

The MB value shall be reported to the nearest 0.01 g of dye per 100 g of the processed sample.

A2.8. Precision and Bias

A2.8.1 Precision—A complete interlaboratory study has not been completed for this test method, therefore no precision information is available at this time.

A2.8.2 Bias—Bias cannot be determined because no material having an accepted reference value is available.

A3. TOTAL ORGANIC CARBON CONTENT OF LIMESTONE

A3.1. Scope

A3.1.1 This annex specifies the laboratory procedures for the quantitative determination of the total organic carbon content of limestone for use as an ingredient in blended cement.

A3.1.2 The text of this annex references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the annex.

A3.1.3 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this annex.

A3.1.4 *This annex 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.*

A3.2. Summary of Test

A3.2.1 Total organic carbon is determined either on a sample from which the inorganic carbon has been removed through hydrochloric acid extraction or by difference of inorganic carbon from total carbon.

A3.3. Significance and Use

A3.3.1 This annex provides a means to determine the total organic carbon content of a sample of finely ground limestone. The organic carbon content of a limestone used as an ingredient in cement may increase the air-entraining agent dosage required to achieve a specific air content in concrete. The specification places a limit on the total organic carbon content of limestone permitted as an ingredient in blended cement.

A3.4. Apparatus

A3.4.1 The equipment shall meet the requirements of Specification C511, unless otherwise specified.

A3.4.2 The following equipment shall be included to perform this test:

- (a) analytical balance, precision = 0.1 mg;
- (b) beaker, 800 mL;
- (c) vacuum flask, 1000 mL;
- (d) glass frit, 40 mm diameter, and porosity = G4;
- (e) magnetic stirrer;
- (f) magnetic stir bar;
- (g) oven, able to maintain a temperature of $45 \pm 5^\circ\text{C}$;
- (h) mortar and pestle made of agate;
- (i) induction furnace equipped with carbon detection; and
- (j) thermogravimetric analyzer.

A3.5. Reagents and Materials

A3.5.1 The following materials shall be included to perform this test:

- (a) hydrochloric acid, analytical grade, $w(\text{HCl}) = 32\%$;
- (b) nitric acid, analytical grade, $w(\text{HNO}_3) = 65\%$;
- (c) silver nitrate solution, $c(\text{AgNO}_3) = 2\text{ mol/L}$;
- (d) gas sources as required for the analyzers, (i.e., oxygen, air, or nitrogen); and
- (e) demineralized water.

A3.6. Sample Preparation

A3.6.1 The sample shall be dried at $45 \pm 5^\circ\text{C}$ and prepared to a pass through a 150- μm (No. 100) sieve conforming to the requirements of Specification E11.

A3.7. Methods to Determine Carbon

A3.7.1 *Methods to Determine Organic Carbon*—Determination of organic carbon shall be conducted in accordance with one of the following options:

(a) Remove the inorganic carbon in accordance with A3.7.3, followed by determining the remaining organic carbon directly in accordance with A3.8; or

(b) Determine the inorganic carbon in accordance with A3.7.2 and, on a separate sub-sample, determine the total carbon in accordance with A3.8. The organic carbon will be calculated by the difference between the total carbon and the inorganic carbon.

A3.7.2 *Determination of Inorganic Carbon by Thermogravimetric Analysis*—This method involves the determination of sample mass loss at various temperatures. The heating of a sample through a temperature range allows for mass loss differentiation based on mineral form (for example, CaCO_3 , MgCO_3 , $\text{Ca}(\text{OH})_2$, for example). Specific operational information is provided by the equipment manufacturers. If free carbon is present, an inert atmosphere (for example, nitrogen) shall be used for sample analysis. The inorganic carbon shall be calculated from the total carbon dioxide released during the analysis. The inorganic carbon as determined shall be recorded as T_i . The total organic carbon shall be calculated in accordance with Eq A3.1.

A3.7.3 *Removal of Inorganic Carbon by Hydrochloric Acid Extraction*—Inorganic carbon shall be removed from the sample by hydrochloric acid extraction as follows:

(a) Pour 100 mL of demineralized water and 80 mL of hydrochloric acid, at 10 mL intervals, into a beaker. Add a magnetic stir bar and stir.

(b) Add 2 g of the sample, weighed to the nearest 0.1 mg, to the diluted hydrochloric acid.

(c) After the carbon dioxide evolution has stopped, add 100 mL of demineralized water and stir the solution for 2 hours.

(d) Filter the solution through a tared glass frit, which was previously dried at $105 \pm 5^\circ\text{C}$. The filtrate must be clear. Filter the filtrate again if it is still cloudy.

(e) Wash the residue with demineralized water till a negative chloride reaction occurs. Control with nitric acid and silver nitrate.

(f) Dry the glass frit with the residue in the oven at a temperature of $45 \pm 5^\circ\text{C}$ to its constant mass.

(g) Pulverize the residue in the mortar and use for further analysis.

(h) Analyze for carbon in the residue in accordance with one of the options detailed in subsection A3.8.

(i) Calculate the total inorganic carbon in accordance with Eq A3.2 and A3.3.

NOTE A3.1—The residue must be free of residual hydrochloric acid. Hydrochloric acid fumes can damage the inner parts of the induction furnace.

A3.8. Determination of Carbon by Instrumental Methods

A3.8.1 *General*—These instrumental methods will report the carbon directly. If the sample being tested has previously had the inorganic carbon removed through acidification, the resulting value will correspond to the result C_I as listed in Eq A3.3. If the sample being analyzed has not previously had the inorganic carbon removed, the resulting value will correspond to T_c as listed in Eq A3.1.

A3.8.2 *Induction Furnace and Infrared Absorption*—This method shall use volatilization of the sample by induction furnace and detection of carbon by infrared absorption. Suitable calibration standards (for example, calcium carbonate and synthetic carbon) are available from some instrument manufacturers. NIST cement SRMs with known additions of NIST argillaceous limestone (or other suitable standards) should also be considered to check instrument calibration.

NOTE A3.2—See Test Methods E1019 for more information on the determination of carbon by the combustion instrumental measurement method.

A3.8.3 *Combustion Gravimetric Method*—The combustion gravimetric method, performed in accordance with ASTM E350, is suitable for the determination of carbon in concentrations from 0.05 to 1.80 % by mass (as carbon dioxide in concentrations from 0.18 to 6.60 % by mass). In this Test Method, the sample shall be burned in a stream of oxygen. The carbon dioxide in the evolved gases shall then be collected in a suitable absorbent and its mass determined. Time of analysis shall be less than 10 min.

NOTE A3.3—See ASTM E350 for more information on the determination of carbon by the combustion gravimetric method.

A3.9. Calculation and Reporting

A3.9.1 The total organic carbon shall be reported to the nearest 0.1 % by mass.

A3.9.2 *Total Organic Carbon Calculation by Difference*—The total organic carbon shall be calculated by subtracting the inorganic carbon determined in accordance with subsection A3.7.2 from the total carbon as determined by one of the options detailed in subsection A3.8 as follows:

$$TOC = T_c - T_i \quad (\text{A3.1})$$

where:

TOC = total organic carbon, % by mass,

T_c = total carbon determined on the sample (subsection A3.8), % by mass, and

T_i = total inorganic carbon determined on the sample (subsection A3.7.2), % by mass.

A3.9.3 *Total Organic Carbon Calculation by Decarbonation Method*—The insoluble residue shall be calculated as follows:

$$R = ((m_3 - m_2) \times 100) / m_1 \quad (\text{A3.2})$$

where:

R = insoluble residue, % by mass,

m_1 = sample weight, g,

m_2 = glass frit empty, g, and

m_3 = glass frit with insoluble residue, g.

The total organic carbon shall be calculated as follows:

$$TOC = (C_I \times R) / 100 \quad (\text{A3.3})$$

where:

TOC = total organic carbon, % by mass,

R = insoluble residue, % by mass, and

C_I = carbon content of the hydrochloric acid treated sample, % by mass.

A3.10. Precision and Bias

A3.10.1 *Precision*—A complete interlaboratory study has not been completed for this test method, therefore no precision information is available at this time (see Note A3.4).

NOTE A3.4—CSA A3004-D2, upon which this procedure is based, states that for a properly conducted decarbonation test:

(a) The detection limit has been found to be 0.02 % by mass carbon.

(b) The limit of quantitation has been found to be 0.05 % by mass carbon.

A3.10.2 *Bias*—Bias cannot be determined because no material having an accepted reference value is available.



APPENDIX

(Nonmandatory Information)

X1. TEST OF CONFORMITY IN RELATION TO A SPECIFIED MB VALUE

X1.1 A check on conformity with a specified methylene blue value can be carried out by making a single addition of dye solution. The volume of dye solution to be injected at one time is given in the following equation:

$$V_2 = (MB_1 \times M_1 + V_1) \quad (X1.1)$$

where:

- V_2 = volume of dye solution to be injected, in mL,
 M_1 = the mass of the test portion, in g,
 MB_1 = the specified MB value, in g of dye per 100 g of the processed sample, and
 V_1 = the volume of dye solution in mL adsorbed by any added kaolinite.

X1.2 After preparation of a test portion, the suspension should be prepared using the test portion, the water and, if necessary, the kaolinite, but including the volume of dye solution. The stain test should be carried out after stirring the suspension for 8 min at 400 ± 40 r/min. If the stain test is positive, the limestone can be assumed to conform to the specification. If this stain test is negative however, the complete determination described in **Annex A2** should be carried out.

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