



Standard Test Method for Portland-Cement Content of Hardened Hydraulic-Cement Concrete¹

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1. Scope

1.1 This test method covers the determination of portland-cement content of a sample of hardened hydraulic-cement concrete.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information purposes only.

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.* Disposal of some or all of the chemicals used in this method may require adherence to EPA or other regulatory guidelines.

2. Referenced Documents

2.1 ASTM Standards:

- C 42 Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete²
- C 114 Test Methods for Chemical Analysis of Hydraulic Cement³
- C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials³
- C 823 Practice for Examination and Sampling of Hardened Concrete in Constructions²
- C 856 Practice for Petrographic Examinations for Hardened Concrete²
- D 1193 Specification for Reagent Water⁴
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes²
- E 832 Specification for Laboratory Filter Papers⁵

3. Significance and Use

3.1 This test method consists of two independent procedures: an oxide-analysis procedure that consists of two sub-

procedures and an extraction procedure. Each procedure requires a substantial degree of chemical skill and relatively elaborate chemical instrumentation. Except for the influence of known interferences, determined cement contents are normally equal to, or slightly greater than, actual values except for the Maleic Acid procedure where results can also be significantly low when the paste is carbonated (Note 1).

NOTE 1—With certain limitations, the procedure is also applicable for estimating the combined content of portland cement and pozzolan or slag in concretes made with blended hydraulic cement and blends of portland cement with pozzolans or slags. The results of this test method when applied to concretes made with blended cements or pozzolans depend on the composition of the pozzolan, the age of the concrete, the extent of reaction of the pozzolan and the fact that this test method may determine only the portland-cement component of a blended cement. The test method should be applied to determination of the blended cement content or the pozzolanic content only by use of calibration concrete samples or other information. Earlier versions of this test method can provide useful information as detailed by Hime⁶ and Minnick.⁷

4. Interferences

4.1 Many constituents of concrete may interfere with the analysis of the concrete for portland-cement content. The following limited lists of materials have been provided as a guide. The rocks, minerals or mineral admixtures listed will interfere with the cement content determination to the extent of their solubility during the dissolution procedure used. The solubility of rocks, minerals or mineral admixtures may depend on the fineness of the test sample, the water-cement ratio of the concrete, the extent of hydration, and the age of the concrete (extended exposure to the high pH of the concrete may affect the solubility of some minerals).

4.2 Substances Affecting Calcium Oxide Sub-procedure:

4.2.1 The following are soluble in even the cold dilute hydrochloric acid of this procedure and will contribute a high bias to the cement content calculated from the soluble calcium oxide: limestone, marble, dolomitic limestone, calcareous

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² *Annual Book of ASTM Standards*, Vol 04.02.

³ *Annual Book of ASTM Standards*, Vol 04.01.

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

⁵ *Annual Book of ASTM Standards*, Vol 15.09.

⁶ Hime, W. G., "Cement Content," *Significance of Tests and Properties of Concrete and Concrete-Making Materials*, ASTM STP 169B, ASTM, 1978, pp. 462–470, and "Analyses for Cement and Other Materials in Hardened Concrete," Chapter 29, *Significant of Tests and Properties of Concrete and Concrete-Making Materials*, ASTM STP 169C, 1994, pp. 315–319.

⁷ Minnick, L. J., "Cement-Content, Hardened Concrete," *Significance of Tests and Properties of Concrete and Concrete-Making Materials*, ASTM STP 169A, ASTM, 1966, p. 326–329.

sandstone, calcareous chert, and caliche encrusted and calcite or dolomite coated rocks.

4.2.2 The following may be soluble depending on the age and pH of the concrete; whether the mineral present is glassy or crystalline, or weathered or strained; and the fineness of the mineral present, and, if soluble, will bias the cement content calculated from the soluble calcium oxide high depending on the calcium content of the minerals: weathered or altered plagioclase feldspar, caliche-encrusted rocks, altered volcanic rocks (with calcareous inclusions), and many other calcium containing rocks.

4.2.3 Every percent of soluble calcium oxide that is contributed by soluble aggregate or mineral admixtures will bias the cement content high by approximately 1.6 %.

4.2.4 Silica fume may lower the acid solubility of the sample and hence bias the result low.

4.3 *Substances affecting the Soluble Silica Sub-procedure:*

4.3.1 The following may be soluble depending on the age and pH of the concrete; whether the aggregate is glassy or crystalline, or weathered or strained; and the fineness of the mineral: chert, opal, chalcedony, glassy volcanic rock, strained quartz (highly strained), quartzite, cataclastic rocks (mylonite, phyllonite), gneiss, schist, metagraywacke, and many other soluble silicon containing rocks or minerals.

4.3.2 Every percent of soluble SiO₂ contributed by aggregates or mineral admixtures will bias the reported cement content high by approximately 4.7 %.

4.3.3 Silica fume may lower the acid solubility of the sample and hence bias the result low. If the digestion time or temperature are sufficient to digest all of the portland cement, the silica fume will also be solubilized and bias the calculated cement content high.

4.4 *Substances affecting the Maleic Acid Procedure:*

4.4.1 The same substances that are soluble in the soluble calcium or the soluble silica subprocedures may be soluble in the maleic acid procedure. (See 4.2.1, 4.2.2 and 4.3.1.)

4.4.2 Every 1 % of the sample that is aggregate or mineral admixture dissolved by the maleic acid will bias the cement content high by 1 %.

4.4.3 Carbonated cement paste may not be soluble in the maleic acid-methanol dissolution and thus may bias the cement content results low.

4.4.4 The unhydrated iron and aluminum phases of the portland cement may not be soluble in the maleic acid and, if not soluble, will bias the cement content low. This may be significant at early ages and less significant at later ages.

5. Apparatus

5.1 Choose the apparatus from applicable items given in Test Methods C 114 and from the following:

- 5.1.1 Chipmunk (jaw ore crusher).
- 5.1.2 Disk Pulverizer.
- 5.1.3 Rotary Mill (rotating puck).
- 5.1.4 Sieve, 300 um (No. 50), 1.18-mm (No. 16) and 4.75-mm (No. 4).
- 5.1.5 Ice Bath or electric cooling apparatus.
- 5.1.6 Steam Bath.
- 5.1.7 Funnel, Buchner-type porcelain funnel.
- 5.1.8 Filter Paper, Type II, Class F and Class G as described

in Specification E 832.

5.1.9 Beakers, 1000 and 250 mL.

5.1.10 Magnetic stirrer, variable speed, with a TFE-fluorocarbon-coated magnetic stirring rod, or an overhead stirrer with a propeller.

5.1.11 Volumetric flask, 1000 mL and 500 mL.

5.1.12 Filtering flask, 2000 mL.

5.1.13 Vacuum pump.

5.1.14 Watch glass, 125 mm.

6. Reagents and Materials

6.1 *Soluble Silica Sub-procedure:*

6.1.1 *Hydrochloric Acid*, reagent grade, density 1.19 Mg/m³.

6.1.2 *Hydrochloric Acid (1:3)*—Mix 300 mL of hydrochloric acid into 900 mL of water.

6.1.3 *Hydrochloric Acid (1:9)*—Mix 100 mL of hydrochloric acid into 900 mL of water.

6.1.4 *Sodium Hydroxide (10 g/L)*—Dissolve 5 g of reagent grade sodium hydroxide in 200 mL of water and dilute to 500 mL.

6.1.5 *Hydrofluoric Acid*, 48 %, reagent grade.

6.1.6 *Sulfuric Acid*, density 1.84 Mg/m³, reagent grade.

6.2 *Calcium Oxide Sub-procedure*—Use reagents as required in Test Methods C 114.

6.3 *Maleic Acid Procedure:*

6.3.1 *Maleic acid*, technical grade.

6.3.2 *Methanol*, technical grade, anhydrous.

6.3.3 *Maleic acid solution*—prepare a fresh solution of 15 % maleic acid in methanol by dissolving and diluting 180 + 1 g of maleic acid with methanol to a final solution volume of 1200 millilitres. Prepare this solution fresh daily. Care must be taken to use methanol only in well ventilated areas, preferably under a hood, to avoid skin contact and breathing vapors. Disposal of the maleic acid/methanol solution shall be according to applicable regulations.

6.3.4 *Fuller's earth*—a clay-like material consisting of a porous colloidal aluminum silicate. Its high adsorptivity has been found very beneficial for decolorizing and purifying materials.

6.4 *Water*—All references to water shall be understood to mean reagent water Types I through IV of Specification D 1193.

7. Sampling

7.1 Choose the concrete sample in accordance with the purposes of the investigation (Note 2).

NOTE 2—A standard procedure for sampling hardened concrete is given in Practice C 823 and a standard procedure for obtaining cores is given in Test Method C 42.

7.2 Both the sample for cement content and for density shall have a minimum length and diameter of four times the nominal maximum size of the aggregate (Note 3).

NOTE 3—A single concrete core taken through the entire depth of the concrete is ordinarily an appropriate sample. This sample may be sawed or split lengthwise to provide samples for cement content, density, and petrographic examination, provided that the length and thickness of the split samples for cement content and density meet the minimum size specified in 7.2. If the split sample would not meet the minimum size

requirement, perform the density measurement first, and then crush the entire dry sample for cement content determination. The recommended mass of concrete for cement content determination is 4.5 kg (10 lb). This mass should be obtained from more than one core when the concrete depth is small and one core will not supply a mass of 4.5 kg (10 lb). If the concrete sample did not have a mass of 4.5 kg (10 lb) it should be so stated in the final report for the cement content result.

7.3 Crush the sample for cement content determination to pass a 4.75-mm (No. 4) sieve, mix thoroughly, and obtain a representative subsample for analysis by coning and quartering or by riffle splitting. The subsample should have a mass of 0.45 kg (1 lb).

8. Cement Content Procedure

8.1 Oxide Analysis Procedure:

8.1.1 Crush or grind the subsample prepared as described in 7.3 using a chipmunk (jaw ore crusher), a disk pulverizer, or a rotary mill (rotating-puck) device, so that all of the material passes a 300- μm (No. 50) sieve. To minimize production of very fine material, use several passes of the sample through the equipment, removing the portion passing the sieve before regrinding the remainder of the sample. Thoroughly mix by coning ten times from one paper to another.

8.1.2 Dry the crushed or ground material in an oven at 105 to 115°C (220 to 240°F) for 3 h and retain the sample in a sealed container.

8.1.3 Sub-procedure to be used:

8.1.3.1 The soluble silica sub-procedure shall be performed in all cases except where a petrographic examination has indicated there are siliceous aggregates or mineral admixtures that will be soluble in cold hydrochloric acid.

8.1.3.2 The calcium oxide sub-procedure shall also be employed unless the aggregate contains a significant amount of calcareous components.

8.1.4 Soluble Silica Sub-procedure:

8.1.4.1 Introduce 100 mL of dilute hydrochloric acid (1:3) into each of three 250-mL beakers. Cool until within the range of 3 to 5°C (38 to 41°F), using an ice bath or electric cooling apparatus.

8.1.4.2 Weigh a 2 g sample to 0.001 g and slowly, over a 1-min period, add it to the cold hydrochloric acid. Maintain the 3 to 5°C (38 to 41°F) temperature for a 5-min period, and stir the mixture either continuously or at least several times during this period (Note 4).

NOTE 4—Observation of the solution during the introduction of the sample may provide useful information. Considerable effervescence indicates a substantial amount of calcite or carbonated paste. Delayed effervescence suggests a dolomitic aggregate. Lack of effervescence suggests the applicability of the calcium oxide sub-procedure.

8.1.4.3 Decant through a Buchner-type porcelain funnel fitted snugly with two disks of a quantitative filter paper for fine precipitates, Type II, Class G filter paper. Once the filtration has begun, take care so that the mat and accumulated residue do not dry completely until the filtration process is complete. Regulate the suction so as to maintain a rapid rate of dripping during the greater part of the filtration. Retain as much of the residue in the beaker as possible. Wash twice by decantation with hot water. Save the filtrate. Transfer the filter paper from the funnel to the beaker containing the balance of

the residue, being careful that no residue is lost. Add 75 mL of hot sodium hydroxide solution (10 g/L) to the residue while stirring, macerate the filter paper, and digest, covered, on a steam bath for 15 min. During the digestion, occasionally stir the mixture. Filter all solids, and wash twice with hot water until the filtrate is neutral to litmus. Combine the filtrates.

8.1.4.4 The filtrate now contains the silica in the form of silicic acid, either in true solution or in suspension in the hydrochloric acid medium. To ensure analysis of only the soluble silicon, refilter any filtrate that is cloudy. (Allowing the filtrate to stand overnight will usually permit suspended silica to settle.) The soluble silica may be analyzed by either of the following procedures 8.1.4.4.1 or 8.1.4.4.2.

8.1.4.4.1 Analysis of soluble silica by conversion to silicon tetrafluoride with hydrofluoric acid—In the case where the aggregate of the original sample contains substantial amounts of material that yields calcium oxide (CaO) on acid treatment, add 10 mL of hydrochloric acid (density 1.19 Mg/m³) to the solution from 8.1.4.4. Transfer to a suitable beaker, with several rinsings of the filter flask. Evaporate to dryness with great care to minimize spattering, bake at not over 120°C (248°F) for 1 h, moisten with hydrochloric acid (density 1.19 Mg/m³), evaporate and bake again, and take up for filtration in 75 mL of hydrochloric acid (1:3). Heat to boiling, filter through an ashless filter paper, and wash the residue with 50 mL of hot hydrochloric acid (1:9) and then with hot water until the washings are free of chlorides. Determine the silica present in the sample by treatment with hydrofluoric acid and sulfuric acid in accordance with the procedure given in Test Methods C 114.

8.1.4.4.2 Instrumental analysis of soluble silica—Transfer the filtrate from 8.1.4.4 to a 500-mL volumetric flask with several rinsings of the filtration flask and bring the volume in the volumetric flask to 500 mL with water. Analyze the soluble silica by any instrumental method found acceptable for cement analysis in accordance with the performance requirement for rapid methods of Test Methods C 114, provided it can be applied to the filtrate. Suitable instrumental techniques may include atomic absorption or inductively coupled plasma spectroscopy.

8.1.4.5 Calculation—Calculate the cement percentage, C_s , by dividing the percent silica (SiO₂) in the concrete by the percent silica (SiO₂) in the cement, and multiplying by 100. If the cement silica value is unknown, assume 21.0 %.

8.1.5 Calcium Oxide Sub-procedure—Calcium oxide may be determined by either of the following procedures. Omit the determination if it is known that the aggregate contains substantial amounts of calcareous components.

8.1.5.1 Oxalate precipitation of calcium—Using the filtrate from the removal of silica (8.1.4.4), separate the ammonium hydroxide group and then determine the calcium oxide, both in accordance with Test Methods C 114, or proceed as described in 8.1.5.2.

8.1.5.2 Instrumental analysis of calcium oxide—Any calcium method found acceptable for cement analysis in accordance with the performance requirements for rapid methods of Test Methods C 114 may be used, provided that the method may be applied to the filtrate of . Further, the method must be