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Standard Test Methods for Determining the Cement Content of Freshly Mixed Concrete[1]

This standard is issued under the fixed designation C 1078; 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.

$^{\epsilon 1}$NOTE—Editorial corrections were made in March 1992.

1. Scope

1.1 These test methods cover two procedures for determining the cement content of a sample of freshly mixed concrete. These methods are applicable to all freshly mixed portland-cement concrete batches for which calibration can be obtained in advance, except those containing certain aggregates or admixtures, that, when washed over a 150-μm (No. 100) sieve, yield significant and varying amounts of calcium ions in solution under the conditions of the test. The choice of which procedure to use is at the discretion of the user. The environment in which these test methods are used may have some bearing on the choice.

1.2 The values stated in inch-pound units are to be regarded as the standard except in regard to sieve sizes which are in accordance with Specification E 11.

1.3 *This standard does not purport to address all of the safety problems, 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:*

C 94 Specification for Ready-Mixed Concrete[2]
C 127 Test Method for Specific Gravity and Absorption of Coarse Aggregate[2]
C 138 Test Method for Unit Weight, Yield, and Air Content (Gravimetric) of Concrete
C 172 Practice for Sampling Fresh Concrete[2]
C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials[2]
C 1079 Test Methods for Determining Water Content of Freshly Mixed Concrete[2]
E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

3. Summary of Test Methods

3.1 A given mass of freshly mixed concrete is washed with a given volume of water over a nest of sieves. The water is agitated so that the cement and other fine particles washed from the concrete (those particles passing the finest sieve) are uniformly suspended. A constant-volume representative sample of the cement suspension is obtained and diluted with a known volume of nitric acid and water. The diluted sample is agitated, without heat, to dissolve some of the calcium compounds in the cement. The calcium-ion concentration of the resulting solution is determined by manual volumetric titration in Procedure A or instrumental fluorometric determination in Procedure B and is correlated to the cement content of the specimen by a previously developed calibration curve.

4. Significance and Use

4.1 These test methods can be used to determine variability of cement content in a batch of concrete and the variability of cement content between batches of nominally identical concrete. If these test methods are used to indicate concrete uniformity, correct and appropriate sampling procedures must be followed. Sampling procedures are referenced in Section 6 of these test methods.

4.2 The water-cement ratio of a concrete sample can be estimated when these test methods are used in conjunction with Test Methods C 1079.

4.3 These test methods are not applicable to concretes containing certain aggregates, or admixtures, that when washed over a 150-μm (No. 100) sieve yield significant and varying amounts of calcium ions in solution under the conditions of the test. The variation of calcium ions will be apparent when performing the calibration procedure. This variation will result in the inability to achieve less than a 5 % variation in the calibration test results or a linear calibration curve (see 9.4 and 15.4).

5. Purity of Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without

[1] These test methods are under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregates and are the direct responsibility of Subcommittee C09.03.03 on Methods of Testing Fresh Concrete.
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[2] *Annual Book of ASTM Standards*, Vol 04.02.
[3] *Annual Book of ASTM Standards*, Vols 04.02 and 14.02.

[4] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

a. Balance	i. Stirrer
b. Hand scoop	j. Buret, 100 mL
c. Sample tub	k. Pipet, 25 mL
d. Specimen tub	l. Conical Beakers of Erlenmeyer flasks
e. Washing machine	m. Fixed-volume dispenser
f. Sieve nest	n. Carboys
g. Linked pipet, 100 and 125 mL	o. Wash Bottle, 500 mL
h. Automatic pipet, 300 mL	p. Dropping bottle

FIG. 1 Equipment Required for Determining the Cement Content of Freshly Mixed Concrete Using Procedure A

lessening the accuracy of the determination. A comparison of results obtained using the proposed grades with results obtained using reagent grade chemicals shall be made to ensure that the proposed grades are sufficiently pure, unless previous records confirm they are satisfactory.

5.2 *Purity of Water*—Unless otherwise indicated, all references to water shall be understood to mean distilled water or water of equal purity. (The primary use of the distilled water is for reagent preparation.)

6. Sampling

6.1 The sample of concrete from which the cement-content test specimen is taken shall be representative of the entire batch and have a mass of not less than 20 kg. It shall be obtained in accordance with Specification C 94 or Practice C 172. Specification C 94 outlines procedures for sampling for uniformity of concrete produced in truck mixers.

PROCEDURE A—MANUAL VOLUMETRIC TITRATION

7. Apparatus

7.1 The following comprises a recommended minimum selection of apparatus for use in conducting the analysis. Apparatus other than that described in 7.1.1 through 7.1.14 can be used provided they serve the same function.[5] A photograph of an acceptable selection of apparatus is shown in Fig. 1.

7.1.1 *Balance,* having a minimum capacity of 2600 g, and a sensitivity of at least 0.1 g.

7.1.2 *Shovels, Hand Scoops, and Rubber Gloves* as required.

7.1.3 *Sample Tub,* 5-qt (4.7-L) polyethylene tub.

7.1.4 *Specimen Tub,* 2-qt (1.9-L) polyethylene tub.

7.1.5 *Washing Machine,* Domestic portable washing machine. It must have a smooth interior, a side-mounted propeller, and a recirculating pump and hose. The recirculating hose shall be fitted with a T connector and 3 ft (0.9 m) of ¼-in. (6.4 mm) inside diameter, latex tubing to be connected to the linked pipet. The working or rated capacity of the tub shall be 10 gal (37.9 L). The inside dimensions of the washing machine tub shall be large enough to hold a 15 5⁄16 by 12 5⁄16-in. (389 by 313-mm) nest of sieves. The washing machine, nest of sieves, and linked pipets are shown in Fig. 2. The cement suspension tank described in 13.1.5 may be fitted with the T connector and substituted for this washing machine.

7.1.6 *Sieve Nest,* having a rectangular steel frame, 15 5⁄16 by 12 5⁄16 by 8 in. (389 × 313 × 203 mm) with a 300-µm (No. 50) sieve at the bottom and a 4.75-mm (No. 4) sieve at mid-height. The 4.75-mm sieve shall be removable. There shall be an additional separate sieve frame, 4 in. (102 mm) deep, nested below the 300-µm sieve, and having a 150-µm (No. 100) sieve at the bottom. The combination of sieves used for calibration and cement-content testing must be the same. The 150-µm sieve may be omitted for siliceous fine aggregates but shall be used when a calcareous fine aggregate is encountered. (A fine aggregate is regarded as "calcareous" when the results of a chemical analysis show in excess 20 % CaO.) If the cement suspension tank described in 13.1.5 is used, the sieve nest described in 13.1.6 shall be used instead of the nest described in this section.

7.1.7 *Linked Pipets,* 125 ± 10-mL glass pipet with an automatic leveling and overflow device fitted with a three-way tap. To it is attached a 100 ± 10-mL automatic pipet with a three-way tap capable of emptying the smaller pipet's contents through the 125 ± 10-mL pipet without draining the contents out of the overflow device. The reproducibility of the automatic pipet must be ±0.20 %. The linked pipet apparatus is shown in Fig. 2.

7.1.8 *Automatic Pipet,* 300 ± 25-mL automatic glass pipet fitted with a three-way tap. The reproducibility of the automatic pipet must be 0.2 %.

7.1.9 *Magnetic Stirrer,* variable-speed magnetic stirrer with a TFE-fluorocarbon-coated magnetic stirring rod.

7.1.10 *Buret,* 100-mL acrylic body Class A or B with a polytetrafluoroethylene (PTFE) plug. A three-pronged utility clamp, a support base, and rod are required to hold the buret.

7.1.11 *Pipet,* 25-mL volumetric glass, Class A or B pipet.

NOTE 1—A rubber suction bulb is recommended for use during pipetting of the 25-mL sample containing cement.

7.1.12 *Conical Beakers or Erlenmeyer Flasks,* narrow-mouth, glass, conical beakers or Erlenmeyer flasks. One 500-mL and one 800- or 1000-mL capacity.

7.1.13 *Fixed Volume Dispenser,* 5-mL fixed-volume dispenser of polyethylene with a polypropylene measuring chamber.

7.1.14 *Reagent Dispensing Containers,* a 500-mL polyethylene wash bottle, a 30-mL polyethylene dropping bottle, two rectangular aspirator carboys of either 2- or 5-gal (7.6- or 18.9-L) capacity, and amber rubber tubing, ½ in. (12.7 mm) in inside diameter by 20 ft (6.1 m) long.

8. Reagents

8.1 *Ammonium Hydroxide-Ammonium Chloride Buffer Solution* (pH = 10)—Add 142 mL of ammonium hydroxide

[5] Howdyshell, P. A., "Revised Operations Guide for a Chemical Technique to Determine Water and Cement Content of Fresh Concrete," Technical Report M-212, U.S. Army Construction Engineering Research Laboratory, April 1977.

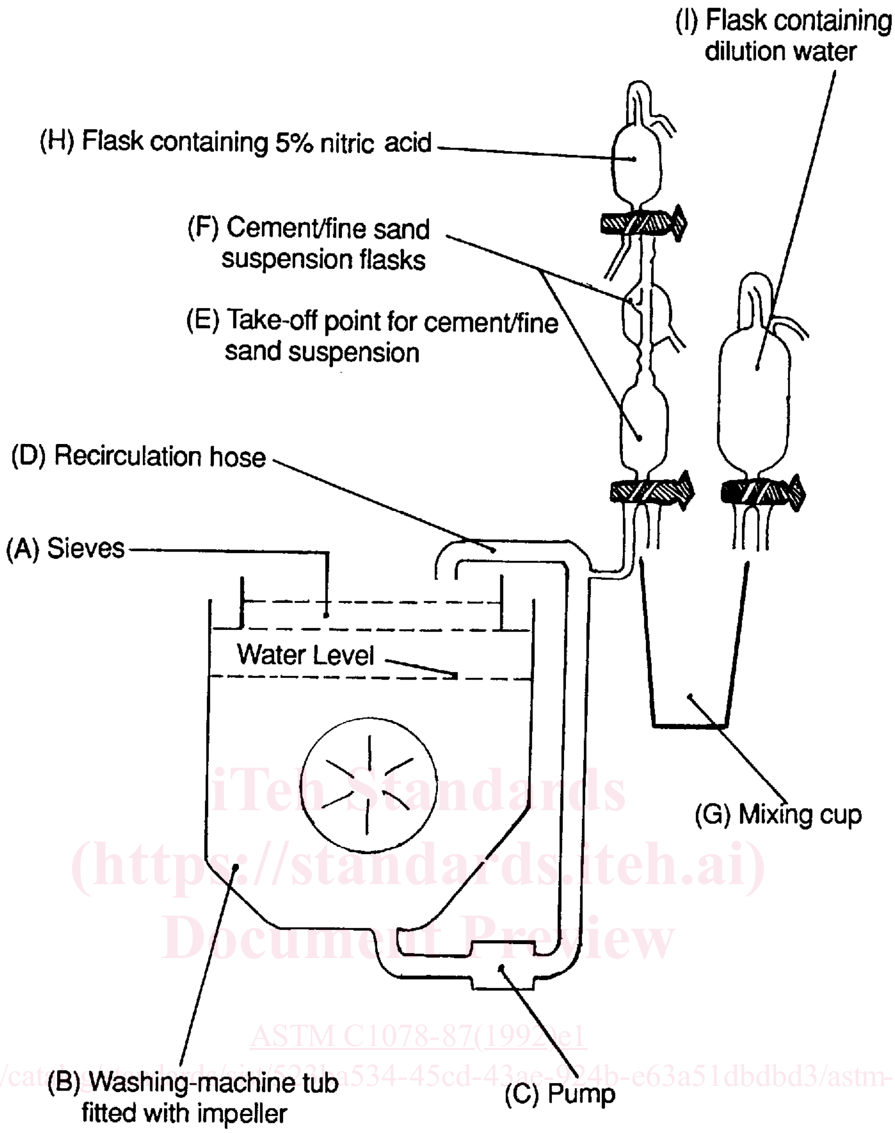

FIG. 2 Equipment for Preparing and Sampling Cement/Fine Sand Suspension Using Procedure A

(NH_4OH) (assay as NH_3 w/w 28–30 %) to 17.5 g of ammonium chloride (NH_4Cl) and dilute to 250 mL with water. The ammonium hydroxide in the buffer solution evaporates rapidly. The solution shall be kept under cover other than when dispensing reagents to minimize evaporation.

8.2 *Eriochrome Black T Indicator Solution*—Dissolve 0.5 g of Eriochrome Black T, Color Index No. 14645, in 25 mL of triethanolamine. The indicator solution has a storage life of about two months.

8.3 *Nitric Acid Solution* (1 + 19)—Add one volume of concentrated nitric acid (HNO_3. sp gr 1.42) to 19 volumes of water.

8.4 *Di-Sodium Ethylenediamine Tetraacetate* (0.01 *M*)—Dissolve 3.72 ± 0.01 g of disodium ethylenediamine tetraacetate (EDTA) in water and dilute to 1 L. Store EDTA solution in polyethylene bottles.

9. Calibration

9.1 *Aggregate Blank*—Prepare a representative 2-kg sample of concrete using the materials and mix proportions of the concrete to be tested minus the cement. The sample mass should weigh 2 kg minus the mass of cement that would be contained in a 2-kg sample of concrete. To determine the relative calcium ion content of these materials follow 10.2 to 10.5. Record the volume of EDTA solution required for the aggregate blank. If 3-kg specimens are tested, the procedure shall be followed for 3 kg instead of 2 kg.

9.2 *Cement*—Using the materials and mix proportions of the concrete to be tested, hand mix a 2.0-kg sample of concrete. (Weigh cement and water for this mix to the nearest gram and weigh aggregates to the nearest 10 g. Make sure all cement from tools, mixing bowl, and the like, is washed into the washing machine.) Determine the relative calcium ion content of this mix by following the procedure in 10.2 to 10.5. Record the volume of EDTA solution required for the cement calibration specimen.

9.3 *Cement Content versus EDTA Calibration Curve*—To construct the linear calibration curve for cement content (g) versus the volume of EDTA required, plot zero cement and the volume of EDTA required for the aggregate blank (9.1) as one point. Plot the mass of cement in the 2-kg concrete calibration sample and the volume of EDTA required (9.2) as the other point. Connect the two points with a straight line to complete the calibration curve for these sources and types of cement and aggregate. All unknown cement contents are linearly proportional in terms of calcium ion content.[6] An example of a calibration curve is in 11.1.

9.4 *Calibration Requirements*—Both the cement calibration test (9.2) and the aggregate blank test (9.1) must be repeated each time tap-water sources additives, aggregate sources, or cement source or type change and every time a new EDTA reagent solution is used. A daily cement calibration test (9.2) shall be made. The results of the three most recent tests must agree within 5 %.[7] If the three most recent tests differ by more than 5 %, three tests shall be made each day. When three successive days pass where the daily test averages are within 5 %, the frequency of the calibration testing may be decreased to one test per day.

10. Procedure

10.1 To obtain the test specimen for concrete containing aggregate sizes up to 1½ in. (38.1 mm), weigh out 2000 ± 200 g of the sample obtained in accordance with 6.1, and record the exact mass of the test specimen to the nearest gram. For concrete containing aggregate sizes greater than 1-½ in. (38.1-mm), weigh out a 3000 ± 300-g test specimen of the sample obtained in accordance with 6.1. Correct for coarse-aggregate variance using the procedure in Section 20 when nominal aggregate size exceeds 1½ in. (correction for smaller aggregate is permitted, if desired).

NOTE 2—A wide range of sample mass is allowed to prevent the mortar-aggregate ratio from being biased through the adjustment of the sample size.

10.2 Fill the washing machine with 10 ± 0.1 gal (37.9 ± 0.4 L) of tap water. Transfer the test specimen to the sieves nested over the washing machine. Start the washing machine's recirculating pump and agitator. Wash the plus 4.75-mm (No. 4) aggregates carefully on the sieve, using the jet of water from the recirculating-pump hose. After all the cement has been removed from the aggregate retained on the 4.75-mm sieve, remove the sieve and the retained material. Wash the plus 300-µm (No. 50) aggregate retained on that size sieve for about 1½ min or until all the cement has been removed from the aggregate. If both the 300-µm and the 150-µm sieves are used, remove the 300-µm sieve at this time and wash the aggregate retained on the 150-µm sieve for 1½ min. Squeeze the end of the large-bore recirculating hose to force the cement suspension to flow through the T connector and the ¼-in. (6.4-mm inside diameter tubing. Rapidly release the large-bore recirculating hose allowing the cement suspension to flow through it. As soon as the hose is released, connect the end of the ¼-in. inside diameter tubing to the 125-mL linked pipet. Squeeze the large-bore recirculating hose again to direct the cement suspension into the pipet. When the pipet is filled to the overflow device, switch off the lower pipet tap and release the large-bore recirculating hose.

10.3 Drain the 125-mL aliquot of cement suspension into the 800-mL beaker. Wash out the 125-mL linked pipet using 100 mL of HNO_3 solution from the automatic pipet positioned above the 125-mL pipet. Leave the tap on the lower 125-mL linked pipet open during this washing so that the acid wash solution will flow into the 800-mL beaker. Dilute the cement-HNO_3 solution in the beaker with 300 mL of tap water from the 300-mL automatic pipet. Place a TFE-fluorocarbon-coated magnetic stirring rod in a beaker and stir contents on a magnetic stirrer for 3 min.

10.4 Pipet off 25 mL of the resulting cement solution after the stirring is completed. Place the 25-mL sample in a 500-mL conical beaker. Using a fixed-volume dispenser, add 10 mL of ammonia-ammonium chloride buffer solution and 4 to 8 drops of the Eriochrome Black T indicator solution from a dropper. The same number of drops shall be used in both the calibration and the test samples. To obtain a clear and distinct end point, the buffer must be added before the indicator solution.

10.5 The calcium-ion concentration of the solution in the 500-mL beaker is determined by an EDTA end-point titration, using a 100-mL buret. Swirl the contents of the beaker during titration. Stop the titration when the solution turns from a red-wine color to a pronounced blue color. Record the volume of the EDTA solution required to reach the end point as V. Operators shall not wear tinted or sun glasses during the calcium titration process, as they can alter the perception of the red-wine to blue end point.

11. Calculation

11.1 Determine the cement content of the sample by referring to the calibration graph.

11.1.1 *Example*—A concrete sample is tested:

Sample mass tested, M_s = 2100 g
EDTA required, V = 30.0 mL

Use the EDTA value on the calibration graph shown in Fig. 3.

11.1.2 From the calibration graph, a 30-mL EDTA corresponds to 300 g of cement (C) in the 2000 ± 200-g sample. The cement content as percent by weight is:

$$\begin{aligned}\text{cement, \%} &= 100\,(C/M_s)\\ &= 100\,(300/2100)\\ &= 14.3\,\%\end{aligned}$$

11.1.3 Cement content in lb/yd³ can be calculated as follows:

$$\text{Cement Content, lb/yd}^3 = (C/M_s)27W$$

where:
C = mass of cement in the test sample, g
M_s = mass of test sample, g, and
W = unit weight of the concrete determined in accordance with Test Method C 138, lb/ft³.

12. Precision and Bias

12.1 Precision—The single operator standard deviation has been found to be 0.9 % cement, percent by weight of the

[6] Neville, A. M., "Properties of Concrete," Pitman Publishing Inc., MA 1981 p. 8.

[7] $\dfrac{\text{(largest cement content)} - \text{smallest cement content)}}{\text{Smallest cement content}} \times 100 \leq 5\,\%$