



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

This standard is issued under the fixed designation C 1084; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method 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 702 Practice for Reducing Samples of Aggregate to Testing Size²

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 and 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:

¹ This method is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.69 on Miscellaneous Tests.

Current edition approved Jan. 10, 2002. Published March 1997. Originally published as C 1084 – 87. Last previous edition C 1084 – 97.

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, *Significance 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.

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 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 SiO_2 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 μm (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/m^3 .

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/m^3 , 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 For cement content determination, crush the sample 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 as described in Practice C 702. 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.

8.1.3.3 All analyses shall be done in triplicate and the average of the three values used in calculating cement content.

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 acceptable for cement analysis in the same silica-present or silica-absent condition as is the case for the state of analysis of the concrete.

8.1.5.3 *Calculation*—Calculate the cement percentage, C_c , by dividing the percentage calcium oxide in the concrete by the percentage calcium oxide in the cement, and multiplying by 100. If the cement calcium oxide value is unknown, assume 63.5 %.

8.2 *Maleic Acid Extraction Procedure:*

8.2.1 Crush or grind and dry the sample as described in 8.1.1 and 8.1.2 except that the material shall pass a 1.18-mm (No. 16) sieve meeting Specification E 11.

8.2.2 *Combined water*—Measure 10 g of the dried crushed sample to the nearest 0.001 g into a tared pre-heated crucible, and dry at $520 \pm 5^\circ\text{C}$ for 3 h or to constant mass (Note 5). Cool in a desiccator and determine the mass.

NOTE 5—The temperature of 540°C should not be exceeded to avoid the decomposition of calcitic or dolomitic carbonates in certain aggregates.

8.2.2.1 Calculate the percentage of combined water as follows:

$$L_c \% = \frac{(C - D) \times 100}{C} \quad (1)$$

where:

L_c = percent combined water, dry basis,

C = mass of dried sample, g, and

D = mass after heating at 520°C , g.

8.2.3 *Extraction*—Determine the mass of a 20-g sample of the ground dried concrete and a 2.5-g sample of Fuller's earth, dried at 105 to 115°C (220 to 240°F), to 0.001 g, and transfer both to a 1000-mL beaker or flask. Add 800-mL of maleic acid solution, and stir with a magnetic stirrer (or overhead stirrer equipped with a propeller) for 60 min. Allow the mixture to settle for 60 min. Filter the solution by carefully decanting through a Whatman 41 filter paper or equivalent of known mass fitted into a 100-mm Buchner funnel, using suction, and collect the filtrate in a 2000-mL vacuum filtering flask. Allow the residue to remain in the beaker.

8.2.3.1 Add 400-mL of maleic acid solution to the residue in the beaker, and stir for 10 min. Allow the mixture to settle for 30 min, and filter the entire contents of the beaker through the original filter paper. Rinse the beaker thoroughly with methanol to insure complete transfer. Wash the residue contained on the filter paper 4 to 5 times with approximately 50 mL portions of methanol to remove any residual maleic acid. Also wash any iron fillings that may be attached to the magnetic stir bar into the Buchner funnel. The filtrate should be clear.

8.2.3.2 Transfer the filter paper and residue onto a 125-mm watch glass, and place in an oven at 105°C (220°F). Dry to a constant mass, and determine the mass of the residue and filter paper to the nearest 0.001 g.

8.2.4 *Calculation*—Calculate the percentage of insoluble residue as follows:

$$R, \% = \frac{(F - G) \times 100}{E} \quad (2)$$

where:

R = percent insoluble residue,

E = mass of sample used, g,

F = combined mass of residue, filter paper, and Fuller's earth, g, and

G = combined mass of filter paper and Fuller's Earth, g.

8.2.5 *Calculation*—Calculate the percent cement content, C_m by subtracting R and L_c from 100.

9. Unit Weight and Loss of Free Water

9.1 Determine the density of a separate sample of the concrete, of a minimum dimension four times the maximum size of the aggregate and a sample mass of at least 0.45 kg (1 lb), as follows:

9.1.1 *Saturated surface dry density*—For saturated surface dry density, which will usually correlate well with the density of the concrete as placed. Immerse the concrete in water and soak it for a minimum of 24 h or to constant weight. (Constant weight may take 48 h for $\frac{1}{2}$ of a 100-mm (4-in.) diameter core.) Determine the mass of the sample while immersed in the water to the nearest 0.1 g (0.001 lb). Remove from the water, surface dry, and determine the mass to the nearest 0.1 g (0.001 lb). Calculate the density, D_{SSD} , in kilograms per cubic metre (or alternately in pounds per cubic foot), as:

$$D_{SSD} = \frac{W_1 \times \rho}{W_1 - W_2} \quad (3)$$

where:

D_{SSD} = saturated surface dry unit weight, kg/m^3 (or alternately lb/ft^3),

W_1 = saturated surface dry (SSD) mass in air after the 48 h soak, kg,

W_2 = SSD mass suspended and immersed in water, kg, and

ρ = density of water, $997 \text{ kg}/\text{m}^3$ ($62.3 \text{ lb}/\text{ft}^3$).

9.1.2 *Dry unit weight and free water*—Dry the sample from 9.1.1 for a minimum of 24 h or to constant weight at 105 to 115°C (220 to 240°F). Cool. Determine the mass to the nearest 0.1 g (0.001 lb). Calculate the density, D_{SSD} , in kilograms per cubic metre (pounds per cubic foot), and the free water, L_f , as:

$$D_{\text{dry}} = \frac{D_{SSD} \times W_3}{W_1} \quad (4)$$

$$L_f = \frac{(W_1 - W_3) \times \rho}{(W_1 - W_2)}$$

where:

D_{SSD} = saturated surface dry unit weight, kg/m^3 (or alternately lb/ft^3),

W_1 = saturated surface dry (SSD) mass in air after the 48 h soak, kg,

W_3 = dry mass, kg, and

L_f = free water, kg/m^3 (or alternately lb/ft^3).

10. Additional Calculations

10.1 *Cement Content*—Calculate the cement content of the

concrete from the data for each procedure used, as follows:

$$\text{Cement content, kg/m}^3 = \frac{C \times D_1}{100} \quad (5)$$

$$\text{Cement content, lb/yd}^3 = \frac{C \times D_2 \times 27}{100} \quad (6)$$

where:

C = determined cement percentage, percent by mass, either as C_s , C_c , or C_m ,

D_1 = D_{dry} , kg/m^3 ,

D_2 = D_{dry} , lb/ft^3 .

11. Report

11.1 Report the cement percentage, and if required, the cement content, to the nearest kg/m^3 (lb/yd^3), as follows:

11.1.1 If two or more procedures or subprocedures have been performed, report the lowest result and report the procedure used.

12. Precision and Bias

12.1 *Precision*—The precision of the cement content test procedures is dependent upon the composition of the concrete.⁸

12.1.1 The use of the soluble silica procedure may lead to erroneously high results for concretes that contain siliceous aggregates, pozzolans, or other cementitious components.

12.1.2 The use of the calcium oxide procedure will lead to erroneously high results if calcareous aggregates or other cementitious materials are present, and may do so with many pozzolanic materials.

12.1.3 The use of the maleic acid procedure may lead to erroneously low or high results, depending upon the solubility of the aggregates in the maleic acid solution, the amount of unhydrated aluminate and ferrite phases in the cement which are insoluble, and the carbonation of the paste.

12.1.4 For these reasons, precision data obtained on one concrete may not be useful in estimating that obtainable for another concrete.

12.1.5 The following sections on the soluble silica and calcium oxide procedures are based on results of cooperative analyses of a suite of three samples (Note 6), one having an aggregate providing a calcium interference, one an aggregate providing a silica interference, and one providing both interferences.

NOTE 6—These precision statements are based on tests of samples that were ground to pass a 600- μm (No. 30) sieve by a single laboratory. For this reason, interlaboratory tests did not include any variation in results that will occur because of sampling and sample preparation.

12.1.6 The section on maleic acid extraction is based on results of cooperative analyses from eleven laboratories on a suite of four concrete samples (Note 7), one with a limestone coarse aggregate, one with dolomite, and two with siliceous aggregates. One specimen contained fly ash. Total cementitious material contents ranged from 250 to 355 kg/m^3 (400 to 600 lb/yd^3).

NOTE 7—This precision statement is based on tests of samples that were ground to pass a 1.18 mm (No. 16) sieve by a single laboratory. For this reason, interlaboratory tests did not include any variation in results that will occur because of sampling and sample preparation.

12.1.7 *Soluble Silica Procedure*—The single laboratory standard deviation has been found to be 15.6 kg/m^3 (26.3 lb/yd^3) (Note 8). Therefore, results of two properly conducted tests in the same laboratory on the same material should not differ by more than 44.1 kg/m^3 (74.3 lb/yd^3) (Note 8).

12.1.7.1 The multilaboratory standard deviation has been found to be 25.1 kg/m^3 (42.3 lb/yd^3) (Note 8). Therefore, results of two properly conducted tests from two different laboratories on samples of the same material should not differ by more than 71.4 kg/m^3 (120.3 lb/yd^3) (Note 8).

12.1.8 *Calcium Oxide Procedure*—Where applicable, the single laboratory standard deviation has been found to be 2.8 kg/m^3 (4.7 lb/yd^3) (Note 8). Therefore, results of two properly conducted tests in the same laboratory on the same material should not differ by more than 7.8 kg/m^3 (13.2 lb/yd^3) (Note 8).

12.1.8.1 The multilaboratory standard deviation has been found to be 10.0 kg/m^3 (16.9 lb/yd^3) (Note 8). Therefore, results of two properly conducted tests from two different laboratories on samples of the same material should not differ by more than 28.4 kg/m^3 (47.9 lb/yd^3) (Note 8).

NOTE 8—These numbers represent, respectively, the 1s and d2s limits as described in Practice C 670.

12.1.9 *Maleic Acid Procedure*—The single laboratory standard deviation has been found to be 14 kg/m^3 (24 lb/yd^3) (Note 8). Therefore, results of two properly conducted tests in the same laboratory on the same material should not differ by more than 40 kg/m^3 (67 lb/yd^3) (Note 8).

12.1.9.1 The multilaboratory standard deviation has been found to be 22 kg/m^3 (37 lb/yd^3) (Note 8). Therefore, results of two properly conducted tests from two different laboratories on samples of the same material should not differ by more than 62 kg/m^3 (104 lb/yd^3) (Note 8).

12.2 *Bias*—The bias of the test procedures is dependent upon the composition of the concrete for reasons described in 12.1.1-12.1.3. For these reasons, bias data obtained on one concrete may be useless in estimating that obtainable for another concrete. Refer to 12.1.5 for a description of the test program on which the following is based.

12.2.1 *Soluble Silica Subprocedure*—For the soluble silica procedure, determined values may be expected to be from about 20 kg/m^3 (50 lb/yd^3) below the true value to 10 kg/m^3 (25 lb/yd^3) above for no silica interference, to 45 kg/m^3 (100 lb/yd^3) or more above for severe silica interferences (for example, for samples containing slag or volcanic aggregate, or a fly ash addition).

12.2.2 *Calcium Oxide Subprocedure*—Where the calcium procedure is applicable, determined values should be within 10 kg/m^3 (25 lb/yd^3) of the true value.

12.2.3 The use of either of the oxide determination procedures may lead to error to the extent that the oxide content used for calculations differs from the true value for the cement. Errors greater than 4 % relative are unusual for this parameter.

12.2.4 Bias may be reduced by analysis of separate samples

⁸ Copies of the research reports used to develop the precision statement are available from ASTM International Headquarters. Request RR:C9-1003 and RR:C9-1012.

of calcareous aggregates representing those used in the concrete, employing the same procedures and applying corrections based on mix design data or estimates of aggregate proportion as based on petrographic studies. Where corrected values are 45 kg/m^3 (100 lb/yd^3) or more below uncorrected values, the method is probably inapplicable.

12.2.5 Maleic Acid Procedure—The bias of the maleic acid procedure is dependent upon the composition of the concrete for reasons stated in 12.1.3. Based on the mean values from the interlaboratory test program described under Precision, the values determined should be within -10 to $+4 \text{ kg/m}^3$ (-17 to $+6 \text{ lb/yd}^3$) of the actual values.

12.2.5.1 Approximately 40 % of the fly ash in the interlaboratory test program was consumed in the extraction and contributed to the determined cement content of the concrete. Other sources have found that variable amounts of fly ash can be consumed in some cases. This demonstrates the need for caution and compensation for induced bias when testing

concretes containing fly ash, slag, silica fume, or natural pozzolans. Various sources of these materials will have highly variable effects on the results from this test.

12.2.5.2 Concrete samples that have deeply carbonated should be tested with caution as the maleic acid test results can be significantly affected, producing erroneously low cement content values.

12.2.6 Particular aggregate sources have been found to significantly affect the bias of this test method. Therefore, the separate testing of aggregate materials may be useful to indicate the solubility of the materials and possible significant contribution from the aggregate to the cement content results.

12.2.7 Petrographic studies such as those performed according to Practice C 856 can be useful in indicating potential interferences to each of the cement content procedures.

13. Keywords

13.1 cement content; concrete; hardened

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