



Standard Test Method for Acid-Soluble Chloride in Mortar and Concrete¹

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^{ε1} NOTE—Editorial changes were made throughout in December 2012.

1. Scope

1.1 This test method² provides procedures for the sampling and analysis of hydraulic-cement mortar or concrete for chloride that is acid soluble under the conditions of test. In most cases, acid-soluble chloride is equivalent to total chloride.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Some values have only SI units because the inch-pound equivalents are not used in practice.

NOTE 1—Sieve size is identified by its standard designation in Specification E11. The alternative designation in parentheses is for information only and does not represent a different standard sieve size.

1.3 The text of this standard references notes and footnotes that provide explanatory information. These notes and footnotes shall not be considered as requirements of this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:³

C42/C42M Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete

C114 Test Methods for Chemical Analysis of Hydraulic Cement

C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

¹ This test 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.

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² This test method is based on a report by Clear, K. C., and Harrigan, E. T., "Sampling and Testing for Chloride Ion in Concrete," Report No. FHWA-RD77-85, Federal Highway Administration, Washington, DC, Aug. 1977 (Available as PB 275-428/AS National Technical Information Services).

³ 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.

C702/C702M Practice for Reducing Samples of Aggregate to Testing Size

C823/C823M Practice for Examination and Sampling of Hardened Concrete in Constructions

C1084 Test Method for Portland-Cement Content of Hardened Hydraulic-Cement Concrete

D1193 Specification for Reagent Water

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Significance and Use

3.1 The amount of acid-soluble chloride in most hydraulic-cement systems is equal to the total amount of chloride in the system. However, some organic substances that may be introduced into mortar or concrete contain chloride that is initially acid-insoluble that can eventually ionize and thus become acid-soluble or water-soluble after a period of exposure in the very alkaline cement system.

3.2 Sulfides are known to interfere with the determination of chloride content. Blast-furnace slag aggregates and cements contain sulfide sulfur in concentrations that can cause such interference and produce erroneously high test results. Treatment with hydrogen peroxide, as discussed in Test Methods C114, is used to eliminate such interference.

3.3 There are aggregates that contain chloride that is not available for corrosion. Such chloride will be detected by the use of this method.⁴

4. Apparatus

4.1 Sampling Equipment

4.1.1 The apparatus required for obtaining samples by coring or sawing is described in Test Method C42/C42M.

4.1.2 Use the following apparatus for sampling by drilling (pulverization):

4.1.2.1 *Rotary Impact Drill* and drill or pulverizing bits of sufficient diameter to provide a representative sample of sufficient size for testing.

⁴ For more information, see "The Determination of the Chloride Content of Concrete," by Brian B. Hope, John A. Page and John S. Poland, *Cement and Concrete Research*, Volume 15, Number 5, Pergamon Press, New York, September 1985, pp. 863-870.

4.1.2.2 *Spoon* or other suitable means to remove pulverized sample material from drill hole without contamination.

4.1.2.3 *Sample Containers* capable of maintaining samples in an uncontaminated state.

4.2 *Sample Processing Apparatus*—The apparatus required for processing samples shall be chosen for its suitability for the purposes of the investigation, and frequently includes a concrete saw and one or more pulverizers.

4.2.1 Samples more than 25 mm (1 in.) in maximum dimension shall be reduced in size by use of a jaw crusher or broken into smaller pieces by hammering carefully to avoid loss of smaller pieces.

4.2.2 Crush particles less than 25 mm (1 in.) in maximum dimension using a rotating puck grinding apparatus, or by using a disk pulverizer, or mortar and pestle operated to restrict to negligible levels the loss of fine particles.

4.2.3 *Sieve*, 850- μ m (No. 20), which shall comply with Specification E11.

4.3 Chloride Determination

4.3.1 *Balance*, shall be capable of reproducing results within 0.0002 g with an accuracy of ± 0.0002 g. Direct-reading balances shall have a sensitivity not exceeding 0.0001 g. Conventional two-pan balances shall have a maximum sensibility reciprocal of 0.0003 g. Any rapid weighing device that may be provided, such as a chain, damped motion, or heavy riders, shall not increase the basic inaccuracy by more than 0.0001 g at any reading and with any load within the rated capacity of the balance.

4.3.2 *Stirrer*, magnetic variable speed, with a TFE-fluorocarbon coated magnetic stirring bar.

4.3.3 *Chloride, Silver/Sulfide Ion Selective Electrode*, or a silver billet electrode coated with silver chloride (see Note 2) with an appropriate reference electrode.

4.3.4 *Potentiometer*, with millivolt scale readable to 1 mV or better. A digital readout is preferred but not required.

NOTE 2—See Note 67 of Test Methods C114 for a discussion of suitable electrodes and coating methods.

4.4 *Glazed Paper*—Paper to which fine particles do not adhere, for use as described in 7.1.

5. 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 Society⁵. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.1.1 *Sodium Chloride* (NaCl).

5.1.2 *Silver Nitrate* (AgNO₃).

5.1.3 *Potassium Chloride* (KCl), (required for silver billet electrode only).

5.1.4 *Reagent Water* conforming to the requirements of Specification D1193 for Type III reagent water.

5.1.5 *Sodium Chloride, Standard Solution* (0.05 N NaCl)—Dry sodium chloride at 105 to 110 °C to a constant mass. Weigh 2.9222 g of dried reagent. Dissolve in water and dilute to exactly 1 L in a volumetric flask and mix thoroughly. This solution is the standard and requires no further standardization.

5.1.6 *Silver Nitrate, Standard Solution* (0.05 N (AgNO₃))—Dissolve 8.4938 g of silver nitrate in water. Dilute to 1 L in a volumetric flask and mix thoroughly. Standardize against 5.00 mL of standard 0.05 N sodium chloride solution diluted to 150 mL with water following the titration test method given in 8.1 beginning with the second sentence. The exact normality shall be calculated from the average of three determinations as follows:

$N = 0.25/V$, where:

N = normality of AgNO₃ solution,

0.25 = milliequivalents NaCl (5.0×0.05 N), and

V = volume of AgNO₃ solution, mL.

Commercially available standard solutions may be used provided the normality is checked according to the standardization procedure.

5.1.7 *Methyl Orange Indicator*—Prepare a solution containing 2 g of methyl orange per litre of 95 % ethyl alcohol.

5.1.8 *Nitric Acid* (1+1).

5.1.9 *Hydrogen Peroxide* (30 %).

6. Sampling

6.1 Select the sample in accordance with Practice C823/C823M or as required for the purpose of the investigation.

6.1.1 Because of the small nominal maximum size of the aggregate in a mortar, pieces of mortar having a mass of 10 g or more will be representative of a rather large volume of mortar.

6.1.2 Take concrete cores in accordance with Test Method C42/C42M unless otherwise specified.

NOTE 3—Concrete cores taken in accordance with Test Method C42/C42M may be cut longitudinally to provide a 12-mm [$\frac{1}{2}$ -in.] thick section generally representative of the core, or cut laterally into 12-mm [$\frac{1}{2}$ -in.] thick disks representative of the concrete core at various depths. Concrete farthest from a surface into which chloride has penetrated often provides chloride data close to that of the originally placed fresh concrete. The cooling water from core cutting may dissolve some of the chloride.

6.1.3 Powdered concrete obtained by use of a rotary impact drill is frequently used in determining chloride concentration with depth in bridge decks, pavements, etc. Such samples may be unrepresentative, especially when the nominal maximum coarse aggregate size is 25 mm (1 in.) or more. Thus, several such samples should be combined, or the data used with care. Procedures for this method of sampling are as follows:

6.1.3.1 Using the rotary impact drill, drill perpendicular to the concrete surface or parallel to the axis of a cored specimen to a specified depth or a depth sufficient to obtain a representative sample of the concrete mixture of at least 20 g of powdered material. To prevent sample contamination, avoid contact of sample with hands and other sources of perspiration.

⁵ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

Clean all sampling tools prior to each sampling operation (See **Note 4**). No lubricants shall be used during drilling.

NOTE 4—Sampling tools may be cleaned with a brush, cloth, ethyl alcohol rinse, water rinse, or other method that will not contaminate the sample.

6.1.3.2 Transfer powdered sample into sample container using a spoon or other suitable means.

7. Sample Preparation

7.1 Pulverize the sample so that all the material will pass a 850- μm (No. 20) sieve. Thoroughly blend the material by coning as described in Practice **C702/C702M** from one glazed paper to another at least 10 times.

8. Procedure

8.1 Determine the mass of approximately 10 g of sample to the nearest 0.01 g and transfer to a 250 mL beaker. Disperse the sample with 75 mL of water. Slowly add 25 mL of dilute (1+1) nitric acid and stir with a glass rod, breaking up any lumps of sample. If the smell of hydrogen sulfide is strongly present, add 3 mL of hydrogen peroxide (See **Note 5**). Add 3 drops of methyl orange indicator to the beaker and stir. Cover the beaker with a watch glass and allow to stand for 1 to 2 minutes. If the solution above the settled solids is not pink colored, add more nitric acid dropwise while stirring until a pink or reddish color persists, then add 10 additional drops of nitric acid and stir. Heat the covered beaker rapidly to boiling and remove from hot plate. Do not allow sample to boil more than a few seconds (See **Note 6**). Make a blank determination using 75 mL of water in place of sample.

NOTE 5—Slags and slag cements contain sulfide sulfur in concentrations that can interfere with chloride determination unless oxidized with hydrogen peroxide.

NOTE 6—To prevent loss of chloride by volatilization, it is important to keep the beaker covered during heating and digestion. Ten seconds of boiling is sufficient. Excessive amounts of acid can erode the silver chloride coating on the silver billet electrode prematurely. A slurry that is only slightly acidic is sufficient.

8.2 Filter the sample through a 9-cm coarse-textured filter paper in a 250 mL or 500 mL Buchner funnel and filtration flask using suction. Rinse the beaker and the filter paper twice with small portions of water. Transfer the filtrate from the flask to a 250 mL beaker and rinse the flask once with water. The original beaker may be used (See **Note 7**). Cool the filtrate to room temperature. The volume should not exceed 175 mL.

NOTE 7—It is not necessary to clean all the slurry residue from the sides of the beaker, nor is it necessary that the filter remove all the fine material. The titration may take place in a solution that contains a small amount of solid matter.

8.3 For instruments equipped with a dial readout it is necessary to establish an approximate "equivalence point" by immersing the electrodes in a beaker of water and adjusting the instrument to read about 20 mV lower than mid-scale. Record the approximate millivoltmeter reading. Remove the beaker and wipe the electrodes with absorbent paper.

8.4 Pipet 2.00 mL of standard 0.05 N NaCl solution into the cooled sample beaker (See **Note 8**). Place the beaker on a magnetic stirrer and add a TFE-fluorocarbon coated stir bar.

Immerse the electrodes into the solution, taking care that the stir bar does not strike the electrodes. Begin stirring gently. Place the delivery tip of the 10 mL buret filled to the zero mark with standardized 0.05 N AgNO_3 in (preferably) or above the sample solution (See **Note 9**).

NOTE 8—It is advisable to maintain constant temperature during measurement, as the solubility of silver chloride varies markedly with temperature at low concentrations.

NOTE 9—If the tip of the buret is out of the solution, any adhering droplet should be rinsed into the beaker with a few millilitres of water following each titration increment.

8.5 Gradually titrate, record the amount of standard 0.05 N silver nitrate solution required to bring the millivoltmeter reading to -60.0 mV of the equivalence point determined in the water.

8.6 Continue the titration with 0.20 mL increments. Record the buret reading and the corresponding millivoltmeter reading. Allow sufficient time between each addition for the electrodes to reach equilibrium with the sample solution (See **Note 10**).

NOTE 10—Experience has shown that acceptable readings are obtained when the minimum scale reading does not change within a 5-s period.

8.7 As the equivalence point is approached, the equal additions of silver nitrate solution will cause larger and larger changes in the millivoltmeter readings. Past the equivalence point, the change per increment will decrease. Continue to titrate until three readings past the approximate equivalence point have been recorded.

8.8 Calculate the precise equivalence point of the titration (See **Note 11**) and subtract the results of the water blank (See **Note 12**)

NOTE 11—An example of recording solution, millivolt readings, and calculation of the titration equivalence point can be found in Appendix X1 of Test Methods **C114**.

NOTE 12—For nonreferee analysis, the blank may be omitted.

9. Calculation

9.1 Calculate percent chloride by mass of mortar or concrete to the nearest 0.001 % as follows:

$$C1, \% = 3.545 [(V_1 - V_2) N]/W, \text{ where:}$$

V_1 = millilitres of 0.05 N AgNO_3 solution used for sample titration (equivalence point),

V_2 = millilitres of 0.05 N AgNO_3 solution used for blank titration (equivalence point),

N = exact normality of 0.05 N AgNO_3 solution,

W = mass of sample, g.

9.2 Other useful measures of chloride concentration can be obtained as follows:

9.2.1 For calculating kilograms of chloride per cubic metre [pounds of chloride per cubic yard] of concrete (see **Note 13**), multiply percent chloride by $D_1/100$ or $D_2/100$ to the nearest 0.1 kg/m^3 [lb/yd^3], where:

D_1 = oven dry density as determined in the section on Concrete Density in Test Method **C1084**, kg/m^3 [lb/yd^3].

D_2 = saturated-surface-dry density as determined in the section on Concrete Density in Test Method **C1084**, kg/m³ [lb/yd³].

Report which density was used in the calculation.

NOTE 13—In the case of drilled samples or where the actual density can not be determined: U = density of mortar or concrete, kg/m³ [lb/yd³]. The air dry density of normal weight concrete for most purposes can be assumed to be 2263 kg/m³ [3815 lb/yd³].

9.2.2 For calculating percent chloride by mass of cement, multiply percent chloride by 100/ P , where:

P = percent cement by mass in the mortar or concrete, as known or determined in accordance with Test Method **C1084**.

9.2.3 For calculating equivalent flake calcium chloride (calcium chloride dihydrate) concentrations, multiply percent chloride by 2.07.

10. Precision and Bias

10.1 Precision⁶

⁶ A copy of the research report used to develop the precision statement is available from ASTM Headquarters. Request RR:C09-1003.

10.1.1 The single laboratory standard deviation has been found to be 0.0015 % chloride (*a*). Therefore, results of two properly conducted tests in the same laboratory on the same material should not differ by more than 0.0042 % (*b*).

10.1.2 The multilaboratory standard deviation has been found to be 0.0021 % (*a*). Therefore, results of two properly conducted tests from two different laboratories on samples of the same material should not differ by more than 0.0059 % (*b*).

NOTE 14—The numbers (*a*) and (*b*) represent, respectively, the (1S) and (D2S) limits as described in Practice **C670**. This precision statement applies to tests of samples prepared and ground by a single laboratory. For this reason, test results will vary depending upon the method of obtaining the sample and the size of the sample before it is crushed and reduced to pass the 850- μ m [No. 20] sieve, or pulverized. The effect may be small for large concrete cores or from mortars sectioned vertically to appreciable for large aggregate concretes sampled by rotary impact drilling with small diameter drills.

10.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, no statement on bias is being made.

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