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Designation: **C 1152/C 1152M – 9703**

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

This standard is issued under the fixed designation C 1152/C 1152M; 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 This test method is under the jurisdiction of ASTM Committee ~~C-9~~ C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.69 on Miscellaneous Tests.

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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 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.

1.3 *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.*

1.4 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system shall be used independently of the other.

2. Referenced Documents

2.1 *ASTM Standards:*³

C 42/C 42M Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete

C 114 Test Methods for Chemical Analysis of Hydraulic Cement

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials

C 702 Practice for Reducing Field Samples of Aggregate to Testing Size

C 823 Practice for Examination and Sampling of Hardened Concrete in Constructions

C 1084 Test Method for Portland-Cement Content of Hardened Hydraulic-Cement Concrete

D 1193 Specification for Reagent Water

E 11 Specification for Wire-Cloth Sieves for Testing Purposes

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 C 114, is used to eliminate such interference.

4. Apparatus

4.1 *Sampling Equipment*

4.1.1 The apparatus required for obtaining samples by coring or sawing is described in Test Method C 42/C 42M.

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.

4.1.2.2 *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- μm [No. 20], which shall comply with Specification E 11.

4.3 ~~The apparatus required for the chloride determination step is given~~

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.

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*, Vol 04.02, volume information, refer to the standard's Document Summary page on the ASTM website.

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 1) 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 1—See Note 67 of Test Methods C 114 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 Society⁴. 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 D 1193 for Type III reagent water.

5.1.5 Sodium Chloride, Standard Solution (0.05 N NaCl)—Dry sodium chloride ~~determination are given~~ 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 det-Merminations~~ three determinations as follows:

$$N = 0.25/V, \text{ where:}$$

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 (14+1).

5.1.9 Hydrogen Peroxide (30 %).

6. Sampling

6.1 Select the sample per Practice C 823 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 C 42/C 42M unless otherwise specified.

NOTE 12—Concrete cores taken in accordance with Test Methods C 42/C 42M may be cut longitudinally to provide a 12-mm [½-in.] thick section generally representative of the core, or cut laterally into 12-mm [½-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 at least 20 g of powdered material. To prevent sample contamination, avoid contact of sample with hands and other sources of perspiration. All sampling tools shall be cleaned and then rinsed with ethyl alcohol and dried prior to each sampling operation. No lubricants shall be used during drilling.

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

⁴ Annual Book Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of ASTM reagents not listed by the American Chemical Society, see Analar Standards, Vol. 04.01, 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.

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 C 702 from one glazed paper to another at least 10 times.

8. Procedure

8.1 Select a sample having a mass of approximately 10 g. Determine the mass of the sample to the nearest 0.01 g and introduce into a 250-mL beaker. Use the reference method for chloride given in Test Methods C 114.

9. Calculation

9.1 Calculate percent chloride by mass of mortar or concrete, by concrete to the reference method nearest 0.001 % as follows:
 $C_1, \% = 3.545 [(V_1 - V_2) N] W$, where:

V_1 = millilitres of 0.05 N AgNO₃ solution used for chloride given in Test Methods C 114, sample titration (equivalence point),

V_2 = millilitres of 0.05 N AgNO₃ solution used for blank titration (equivalence point),

N = exact normality of 0.05 N AgNO₃ 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 3), multiply percent chloride by ~~$U/100$~~ , $D_1/100$ or $D_2/100$ to the nearest 0.1 kg/m^3 [lb/yd^3], where:

U = density of mortar or concrete, oven dry density as determined in the section on Concrete Density in Test Method C 1084, kg/m^3 [lb/yd^3]. The air-dry.

D_2 = saturated-surface-dry density of normal weight concrete for most purposes can be assumed to be 2263 as determined in the section on Concrete Density in Test Method C 1084, kg/m^3 [lb/yd^3].

Report which density was used in the calculation.

NOTE 3—In the case of drilled samples or where the actual density can not be determined: U = density of mortar or concrete, kg/m^3 [lb/yd^3]. The air dry density of normal weight concrete for most purposes can be assumed to be 2263 kg/m^3 [3815 lb/yd^3].

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 C 1084.

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 Precision⁵

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 24—The numbers (a) and (b) represent, respectively, the (1S) and (D2S) limits as described in Practice C 670. 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- μm [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.

Annual Book

⁵ A copy of the research report used to develop the precision statement is available from ASTM Standards, Vol 14.02, Headquarters. Request RR:C9-1003.



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