
Standard Method of Test for

Sampling and Testing for Chloride Ion in Concrete and Concrete Raw Materials

AASHTO Designation: T 260-21

Technically Revised: 2021

Editorially Revised: 2021

Technical Subcommittee: 3c, Hardened Concrete



**American Association of State Highway and Transportation Officials
555 12th Street NW, Suite 1000
Washington, DC 20004**

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1. SCOPE

- 1.1. This method covers procedures for the determination of the acid-soluble chloride ion content or the water-soluble chloride ion content of aggregates, portland cement, mortar, or concrete.
- 1.2. The total amount of chloride is usually equal to the acid-soluble chloride. However, organic additives or minerals that contain acid-insoluble chloride may be present in concrete and concrete raw materials. These constituents may become acid soluble during long-term exposure to the alkaline environment in concrete or mortar.
- 1.3. The age of concrete mortar or hydrated portland cement at the time of sampling will have an effect on the water-soluble chloride ion content. Therefore, unless early age studies are desired, it is recommended that the material be well cured and at least 28 days of age before sampling.
- 1.4. This standard provides for the determination of chloride ion content by two procedures: Procedure A, Determination of Acid-Soluble Chloride Ion Content and Water-Soluble Chloride Ion Content by Potentiometric Titration or Ion-Selective Electrode (Laboratory Test Method); and Procedure B, Acid-Soluble Chloride Ion by Atomic Absorption (Laboratory Test Method).
- 1.5. 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, which can be eliminated by treatment as noted in the test procedures. Other materials that produce a strong odor of H₂S when acid is added to them should be similarly treated.
- 1.6. The values stated in SI units are to be regarded as the preferred standard.
- 1.7. *The quality of the results produced by this standard are dependent on the competence of the personnel performing the procedure and the capability, calibration, and maintenance of the equipment used. Agencies that meet the criteria of R 18 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with R 18 alone does not completely assure reliable results. Reliable results depend on many factors; following the suggestions of R 18 or some similar acceptable guideline provides a means of evaluating and controlling some of those factors.*

2. REFERENCED DOCUMENTS

- 2.1. *AASHTO Standards:*
 - M 231, Weighing Devices Used in the Testing of Materials

- R 18, Establishing and Implementing a Quality Management System for Construction Materials Testing Laboratories
- R 71, Sampling and Amount of Testing of Hydraulic Cement
- R 76, Reducing Samples of Aggregate to Testing Size
- R 90, Sampling Aggregate Products

2.2. *ASTM Standard:*

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

2.3. *Other Documents:*

- Clear, K. C. *Evaluation of Portland Cement Concrete for Permanent Bridge Deck Repair*. Report FHWA-RD-74-5. Federal Highway Administration, U.S. Department of Transportation, February 1974.
- Clemena, G. C., J. W. Reynolds, and R. M. McCormick. *Comparative Studies of Chloride in Hardened Concrete*. Report No. FHWA-RD-77-84 77-R7. Virginia Highway and Transportation Research Council, August 1976.

PROCEDURE A—ACID-SOLUBLE CHLORIDE ION CONTENT AND WATER-SOLUBLE CHLORIDE ION CONTENT BY POTENTIOMETRIC TITRATION OR ION-SELECTIVE ELECTRODE (LABORATORY TEST METHOD)

3. APPARATUS

3.1. *Sampling Equipment for Procedures A and B:*

- 3.1.1. Core drill.
- 3.1.2. Rotary impact-type drill with a depth indicator and drill or pulverizing bits of sufficient diameter to provide a representative sample of sufficient size for testing.
 - 3.1.2.1. Sample containers capable of maintaining the sample in an uncontaminated state.
 - 3.1.2.2. Spoons of adequate size to collect the sample from the drilled holes.
 - 3.1.2.3. A “blow out” bulb or other suitable means of removing excess pulverized material from the hole prior to redrilling operations.
 - 3.1.2.4. A device capable of determining the location and depth of steel reinforcement to ± 3 mm ($\pm 1/8$ in.).

3.2. *Equipment for Chemical Testing:*

- 3.2.1. Chloride ion or silver/sulfide ion selective electrode and manufacturer-recommended filling solutions.

Note 1—Suggested electrodes are the Orion 96-17 Combination Chloride Electrode or the Orion 94-6 Silver/Sulfide Electrode or equivalents. The Silver/Sulfide Electrode requires use of an appropriate reference electrode (Orion 90-02 or equivalent).

- 3.2.2. A millivoltmeter compatible with the ion electrode.
Note 2—Suggested millivoltmeter is the Orion Model 701 A Digital pH/mV meter or equivalent.
- 3.2.3. Magnetic stirrer and Teflon stirring bars.
- 3.2.4. Buret with 0.1-mL graduations.
- 3.2.5. Balance complying with M 231, Class A.
- 3.2.6. Balance complying with M 231, Class G 2.
- 3.2.7. Hot plate, 250 to 400°C heating surface temperature.
- 3.2.8. Glassware, 250-mL beakers, filter funnels, stirring rods, watch glasses, dropper, wash bottles.
- 3.2.9. Sieve, U.S. Standard 300 μm (No. 50).
- 3.2.10. Whatman No. 40 and No. 41 filter papers (or equivalent).
Note 3—If equivalent filter papers are used, they should be checked to confirm they do not contain chloride, which will contaminate the sample.

4. REAGENTS

- 4.1. Concentrated HNO_3 (sp gr 1.42).
- 4.2. Sodium chloride, NaCl , reagent grade (primary standard).
- 4.3. Standard 0.01 normality NaCl solution. Dry reagent grade NaCl in an oven at 105°C. Cool, in a desiccator, determine the mass of approximately 0.5844 g to the nearest 0.0001 g, dissolve in distilled H_2O , and transfer to a 1-L volumetric flask. Make up to the mark with distilled H_2O and mix. Calculate the exact normality as follows:

$$N_{\text{NaCl}} = (0.0100) \frac{(W_{\text{actual}})}{0.5844} \quad (1)$$

where:

W_{actual} = actual mass of NaCl ; and
 N_{NaCl} = normality of NaCl solution.

- 4.4. *Standard 0.01 Normality AgNO_3* —Determine the mass of 1.7 g of reagent AgNO_3 , dissolve in distilled H_2O , filter into a 1-L brown glass bottle, fill, and mix thoroughly. Standardize against 25.00 mL of the NaCl solution by the titration method given in Section 6.4. Calculate the exact normality as follows:

$$N_{\text{AgNO}_3} = \frac{(V_{\text{NaCl}})(N_{\text{NaCl}})}{V_{\text{AgNO}_3}} \quad (2)$$

where:

N_{AgNO_3} = normality of AgNO_3 solution;
 V_{NaCl} = volume (mL) of NaCl solution;
 N_{NaCl} = normality of NaCl solution; and
 V_{AgNO_3} = volume (mL) of AgNO_3 solution.

- 4.5. *Distilled Water.*

Note 4—Deionized water may be used in place of distilled water for samples where extreme precision and accuracy are not demanded.

- 4.6. *Methyl Orange Indicator.*
- 4.7. *Ethanol*—Denatured, or methanol, technical.
- 4.8. *Hydrogen Peroxide*—30 percent.

5. METHOD OF SAMPLING

5.1. *Concrete Sample:*

- 5.1.1. Determine the depth within the concrete for which the chloride content is desired.

Note 5—A convenient method of determining the location and depth of reinforcement bars is a pachometer capable of determining the location and depth of steel reinforcement to ± 3 mm (0.125 in.).

- 5.1.2. *Core Method*—Drill the core to chosen depth and retrieve.

- 5.1.2.1. When samples are received in the laboratory in other than pulverized condition, the sample shall be crushed and ground to a powder. All sawing or crushing shall be done dry (i.e., without water). All material shall pass a 0.300-mm (No. 50) sieve. All pulverizing tools and sieves shall be washed with alcohol or distilled water and shall be dry before use with each separate sample. (See Note 7.)

5.1.3. *Pulverizing Method:*

- 5.1.3.1. Set the rotary hammer depth indicator so that it will drill to 13 mm (0.5 in.) above the desired depth.
- 5.1.3.2. Using a drill or pulverizing bit, drill until the depth indicator seats itself on the concrete surface.
- 5.1.3.3. Thoroughly clean the drilled hole and surrounding area utilizing the “blow out” bulb or other suitable means.
- 5.1.3.4. Reset the depth indicator to permit 13 mm (0.5 in.) additional drilling.
- 5.1.3.5. Pulverize the concrete until the depth indicator again seats itself on the concrete.

Note 6—Care must be exercised during this pulverizing operation to prevent the drill bit from abrading concrete from the sides of the hole above the sampling depth. To ensure against this, some users utilize a 6-mm (0.25-in.) smaller diameter bit in this step than that used in Section 5.1.3.2.
- 5.1.3.6. Collect at least 10 g of the material remaining in the hole using a spoon and place in the sample container.
- 5.1.3.7. If the sample, as collected, does not completely pass a 0.300-mm (No. 50) sieve, additional pulverizing shall be performed in the laboratory until the entire sample is finer than 0.300-mm (No. 50).

Note 7—During sample collection and pulverizing, personnel shall use caution to prevent contact of the sample with hands, or other sources of body perspiration or contamination. Further, all sampling tools (drill bits, spoons, bottles, sieves, etc.) shall be washed with alcohol or distilled