

Designation: C1524 - 02a (Reapproved 2010)

Standard Test Method for Water-Extractable Chloride in Aggregate (Soxhlet Method)¹

This standard is issued under the fixed designation C1524; 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 provides procedures for sampling and analysis of aggregate for water-extractable chloride using a Soxhlet extractor.

Note 1—This test method is to be used when significantly high chloride content has been found in aggregates, concretes, or mortars.

- 1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in 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.

2. Referenced Documents

2.1 ASTM Standards:²

C114 Test Methods for Chemical Analysis of Hydraulic Cement

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

C1152/C1152M Test Method for Acid-Soluble Chloride in Mortar and Concrete

C1218/C1218M Test Method for Water-Soluble Chloride in Mortar and Concrete

D75 Practice for Sampling Aggregates

D1193 Specification for Reagent Water

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

2.2 American Concrete Institute Standard:

ACI 222.1–96 Provisional Standard Test Method for Water-Soluble Chloride Available for Corrosion of Embedded

Steel in Mortar and Concrete Using the Soxhlet Extractor³

3. Significance and Use

- 3.1 Water-extractable chloride, when present in sufficient amount, has a potential to initiate or accelerate corrosion of metals, such as steel, embedded in or contacting a cementitious system, such as mortar, grout, or concrete. This test method is applicable when aggregates contain a high background of naturally occurring chloride (see ACI 222.1–96). Test Method C1152/C1152M determines acid-soluble chloride and Test Method C1218/C1218M determines water-soluble chloride. Both Test Methods C1152/C1152M and C1218/C1218M pulverize the sample to a fine powder or fine granular material. The Soxhlet method is intended to use nonpulverized material. Results with some aggregates have shown that the Soxhlet procedure extracts an extremely low amount of chloride, with most of it remaining in the rock, and therefore, it is not available for corrosion.
- 3.2 The Soxhlet extraction apparatus consists of three sections: the boiling flask, which contains reagent water at the beginning of the test; the extractor, which contains the sample inside a thimble; and, the condenser. The extractor functions by boiling the water, which condenses and drips on to the sample. When the water attains a fixed height above the sample, the extractor siphons the water from the thimble back to the boiling flask. The process repeats itself until the test is terminated. (Refer to Fig. 1.)

4. Apparatus

- 4.1 Sampling Equipment:
- 4.1.1 The apparatus required for obtaining aggregate samples is described in Practice D75.
- 4.2 *Sampling Processing Apparatus*, the same as in Test Method C1218/C1218M without pulverization.
- 4.3 Soxhlet Extractor Apparatus, shall have the following minimum size and consist of the following (see Fig. 1).
- 4.3.1 *Reflux-Type Condenser*, with a 34/45-mm ground-glass fitting to the extractor, and having a 27-mm inside diameter by a 192-mm body length.

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 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.

³ Available from American Concrete Institute (ACI), P.O. Box 9094, Farmington Hills, MI 48333-9094, http://www.concrete.org.

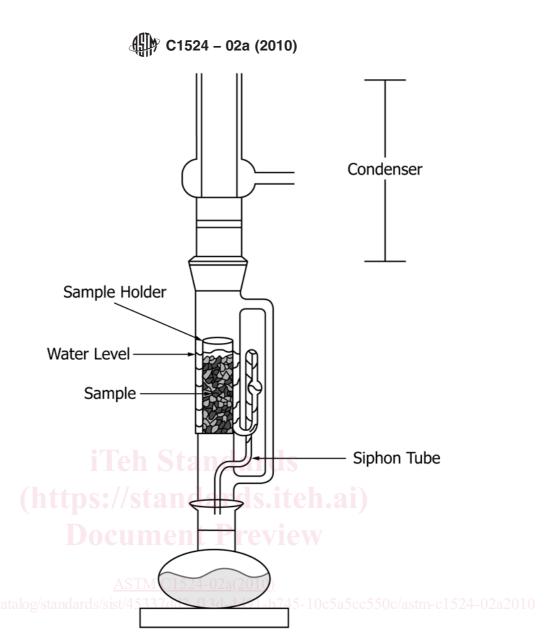


FIG. 1 Soxhlet Extraction Apparatus

- 4.3.2 *Soxhlet Extraction Tube*, with a 34/45-mm ground-glass fitting to the condenser and a 24/40-mm ground-glass fitting to the boiling flask, for holding sample thimbles 25 mm by 80 mm (diameter by height).
- 4.3.3 A 250-mL boiling flask with a 24/40-mm ground-glass fitting.
- 4.3.4 A heating mantel, burner, or hot plate configured for the flask capable of attaining 200°C.
- 4.3.5 *Suitable Sample Holder*, such as a porous extraction thimble having a 25-mm inside diameter and 80-mm external length.
- 4.4 The apparatus required for the chloride determination step is given in the test method for chloride in Test Methods C114.
 - 4.5 pH Paper, short-range 0.0-3.0.
- 4.6 *Drying Oven*, of sufficient size, capable of continuously heating at 110 ± 5 °C.
 - 4.7 Sieve, 25.0 mm, complying with Specification E11.

5. Reagents

- 5.1 The reagents required for the chloride determination are given in the test method for chloride of Test Methods C114, and the Procedure section of Test Method C1218/C1218M.
- 5.2 Reagent water is either deionized or distilled, conforming to the requirements of Specification D1193 for Type III reagent water.

6. Sample Preparation

- 6.1 Preparation:
- 6.1.1 For Nominal Maximum Aggregate Sizes 25.0 mm or Greater: Use a jaw crusher or hammer, and reduce the sample so that the particles pass a 25.0-mm sieve and are representative of the sample. Use a sample splitter or use coning and quartering to reduce the sample to between 200 and 500 g. Do not crush the sample to a powder. Oven dry the reduced sample at $110 \pm 5^{\circ}$ C for 2 h.
- 6.1.2 For Nominal Maximum Aggregate Sizes Less Than 25.0 mm: Use a sample splitter or use coning and quartering to