Designation: C 1524 – 02a

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

This standard is issued under the fixed designation C 1524; 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.

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:*

C 114 Test Methods for Chemical Analysis of Hydraulic Cement[2]

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials[3]

C 1152/C 1152M Test Method for Acid-Soluble Chloride in Mortar and Concrete[3]

C 1218/C 1218M Test Method for Water-Soluble Chloride in Mortar and Concrete[3]

D 75 Practice for Sampling Aggregates[4]

D 1193 Specification for Reagent Water[5]

E 11 Specification for Wire Cloth and Sieves for Testing Purposes[6]

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 Extractor[7]

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 C 1152/C 1152M determines acid-soluble chloride and Test Method C 1218/C 1218M determines water-soluble chloride. Both Test Methods C 1152/C 1152M and C 1218/C 1218M 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 D 75.

4.2 *Sampling Processing Apparatus*, the same as in Test Method C 1218/C 1218M 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.

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[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] *Annual Book of ASTM Standards*, Vol 04.01.

[3] *Annual Book of ASTM Standards*, Vol 04.02.

[4] *Annual Book of ASTM Standards*, Vol 04.03.

[5] *Annual Book of ASTM Standards*, Vol 11.01.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

[7] Available from American Concrete Institute (ACI), P.O. Box 9094, Farmington Hills, MI 48333.

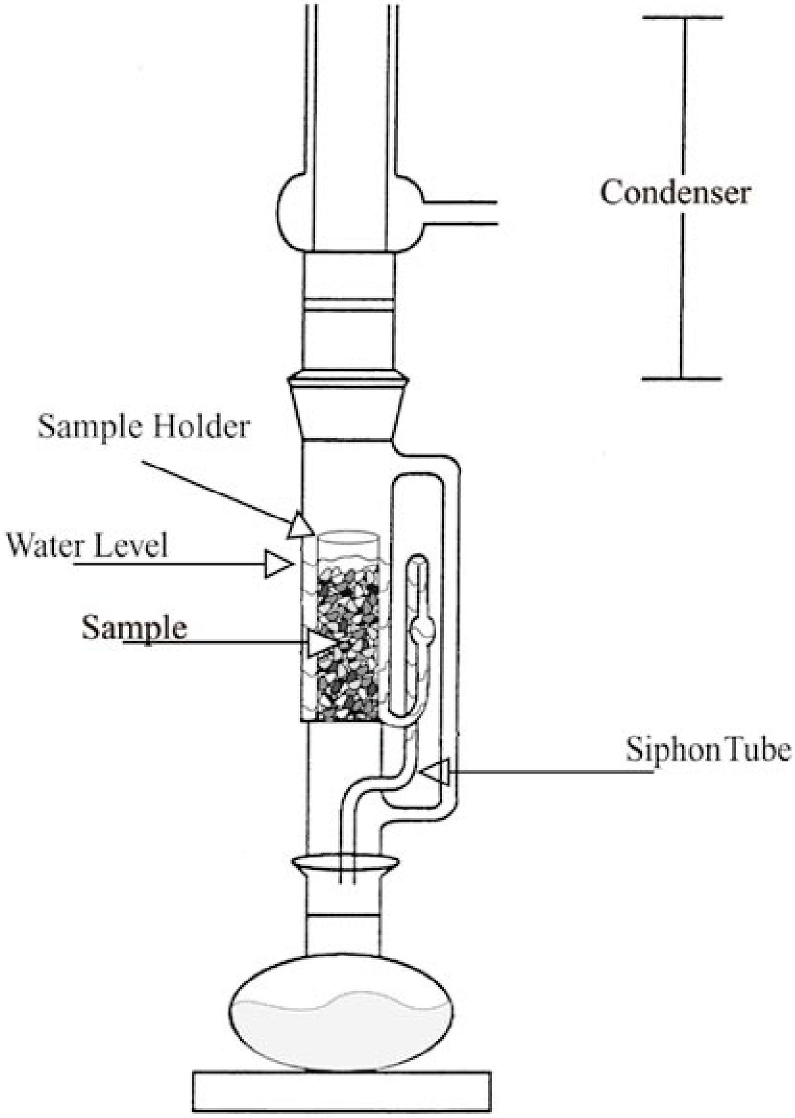

FIG. 1 Soxhlet Extraction Apparatus

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 C 114.

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 E 11.

5. Reagents

5.1 The reagents required for the chloride determination are given in the test method for chloride of Test Methods C 114, and the Procedure section of Test Method C 1218/C 1218M.

5.2 Reagent water is either deionized or distilled, conforming to the requirements of Specification D 1193 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 ± 5°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 reduce the sample to between 200 and 500 g. Do not crush the sample to a powder. Oven dry the reduced sample at 110 ± 5°C for 2 h.

7. Procedure

7.1 Mix and cone the reduced sample three times before sampling each replicate. Obtain a minimum mass of 30.0 g of the sample by sampling from at least four points, record the mass to the nearest 0.01 g, and transfer it to the porous sample holder of the Soxhlet extractor. Place at least 200 mL of reagent water in the lower flask. Conduct this procedure in triplicate. Make a blank determination by using the Soxhlet apparatus, with thimble but containing no sample.

NOTE 2—Replicate sample size can be larger than 30 g; however, the top of the sample in the thimble needs to be kept below the top of the siphon to properly extract the sample with solution. The sample will include aggregate particles and the fines produced by crushing.

7.1.1 Place the top of the thimble above the top of the glass tube regulating the level at which the water siphons off. Do not completely fill the thimble with the sample. Assemble the condenser complete with cooling water supply tubing to the extractor and place on the heater. Turn on both the heater and condenser cooling water and allow extraction to continue for 24 h; the heating rate shall be adjusted to give a cycle every 20 ± 5 min. The number of cycles shall be not less than 70. One cycle consists of the thimble filling with solution and discharging.

NOTE 3—Larger-sized Soxhlet apparatus are available; however, the cycle times will be longer and the total extraction time for 70 cycles will be about 1.5 to 3 days.

7.1.2 At the conclusion of the extraction stage, quantitatively transfer the solution to a 400-mL beaker by rinsing the boiling flask three times with 10 mL of reagent water, and transferring the washings to the beaker. Add 3 ± 0.1 mL of nitric acid that has been diluted one-on-one with water, and add 3 ± 0.1 mL 30 % solution hydrogen peroxide, to the extract. Using pH paper, determine the pH of the solution. If the pH of the solution is more than 2, add additional 1:1 nitric acid until the pH is less than 2. Cover the beaker with a watch glass. Add a stirring bar composed of polytetrafluoroethylene (PTFE) and allow to stand for 1 to 2 min. While stirring, heat the covered beaker rapidly to boiling. Do not boil for more than a few seconds. Remove from the hot plate.

NOTE 4—It is important to keep the beaker covered during heating and digestion to prevent the loss of volatile chloride.

7.1.3 Proceed in accordance with the reference test method for chloride in Test Methods C 114, starting with the procedure that follows removal of the sample from the hot plate.

8. Calculation and Reporting

8.1 Calculate percent chloride by mass of oven-dry aggregate by the reference test method for chloride in Test Methods C 114.

8.2 Report both the individual and average values of Soxhlet extracted chloride content.

8.3 The report shall state whether the sample was fractured or not fractured during preparation for testing.

9. Precision and Bias

9.1 *Precision*:

9.1.1 The single-laboratory standard deviation has been found to be 0.0004 % [8] chloride by mass of aggregate. Therefore, two acceptable test results are not expected to differ by more than 0.0015 %.

9.1.2 The multilaboratory standard deviation has been found to be 0.0006 % [8] chloride by mass of aggregate. Therefore, acceptable results obtained in two different laboratories are not expected to differ by more than 0.0020 %.

9.2 *Bias*—The procedure in this test method has no bias because the value of Soxhlet-extracted chloride is defined by the procedure (see Practice C 670).

10. Keywords

10.1 aggregate; chloride content

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[8] These numbers represent, respectively, the (1s) and (d2s) limits as described in Practice C 670.

APPENDIX

(Nonmandatory Information)

X1. Summary of Soxhlet Round Robin Precision Testing

X1.1 Round robin testing to determine within-laboratory and between-laboratory precision of the Soxhlet method was performed by six laboratories. Each laboratory was provided with three aggregates to analyze, two containing integral chloride and one aggregate containing chloride absorbed from a marine environment. The aggregates were analyzed as received using the Soxhlet method. The results of the interlaboratory study are found in Tables X1.1-X1.3.

NOTE X1.1—Data from Aggregate C was not used to calculate within-laboratory and between-laboratory precision.

X1.2 The Soxhlet method is intended for the analyses of limestone aggregates that contain a large amount of chloride that is encapsulated or integral within the aggregate shell. These are primarily aggregates from the Niagara Escarpment extending from southern Ontario down through New York,

TABLE X1.1 Aggregate A Containing Integral Chloride—Chloride, % by Mass of Sample

Laboratory	Data			Average	Standard Deviation
	a	b	c	X_n	S_n
1	0.0012	0.0021	0.0011	0.0015	0.00055
2	0.0004	0.0014	0.0009	0.0009	0.00050
3	0.0021	0.0023	0.0023	0.0022	0.00012
4	0.0010	0.0008	0.0025	0.0014	0.00093
5	0.0011	0.0012	0.0011	0.0011	0.00006
6[A]	0.0226	0.0226	0.0051	0.0168	0.01010

[A] Data from Laboratory 6 was not used to calculate the between-lab average.

TABLE X1.2 Aggregate B Containing Integral Chloride—Chloride, % by Mass of Sample

Laboratory	Data			Average	Standard Deviation
	a	b	c	X_n	S_n
1	0.0025	0.0028	0.0022	0.0025	0.00030
2	0.0021	0.0024	0.0017	0.0021	0.00035
3	0.0015	0.0018	0.0021	0.0018	0.00030
4	0.0023	0.0021	0.0016	0.0020	0.00036
5	0.0011	0.0010	0.0016	0.0012	0.00032
6[A]	0.0342	0.0301	0.0242	0.0295	0.00503

[A] Data from Laboratory 6 was not used to calculate the between-lab average.

TABLE X1.3 Aggregate C Containing Absorbed Chloride—Chloride, % by Mass of Sample

Laboratory	Data			Average	Standard Deviation
	a	b	c	X_n	S_n
1	0.2279	0.2780	0.2739	0.2766	0.00234
2	0.2301	0.2880	0.2954	0.2712	0.03576
3	0.3201	0.3172	0.3174	0.3182	0.00162
4	0.3065	0.2824	0.2971	0.2953	0.01215
5	0.2704	0.2984	0.2511	0.2733	0.02378
6	0.2088	0.2047	0.2673	0.2269	0.03502

northern Michigan and into the Chicago area. Aggregates of this type will produce a high chloride value when ground and analyzed according to Test Method C 1218/C 1218M and a substantially lower chloride value analyzed by the Soxhlet method.

X1.3 The Soxhlet method is not intended for marine aggregates or aggregates that contain absorbed chloride as indicated by an insignificant difference between water-soluble (Test Method C 1218/C 1218M) and water-extractable (Soxhlet) chloride.