

# Provisional Standard Test Method for Water-Soluble Chloride Available for Corrosion of Embedded Steel in Mortar and Concrete Using the Soxhlet Extractor

Reported by ACI Committee 222

*Some water-soluble chlorides, primarily in certain aggregates, do not induce corrosion of embedded reinforcing steel since these chlorides are bound within the aggregate. Currently, available test methods cannot distinguish between the water-soluble chlorides that support corrosion and those that do not. This test method detects only water-soluble chlorides that contribute to the corrosion of the reinforcing steel.*

**Keywords:** *Water-soluble chlorides; corrosion; steel; mortar; concrete; Soxhlet Extractor.*

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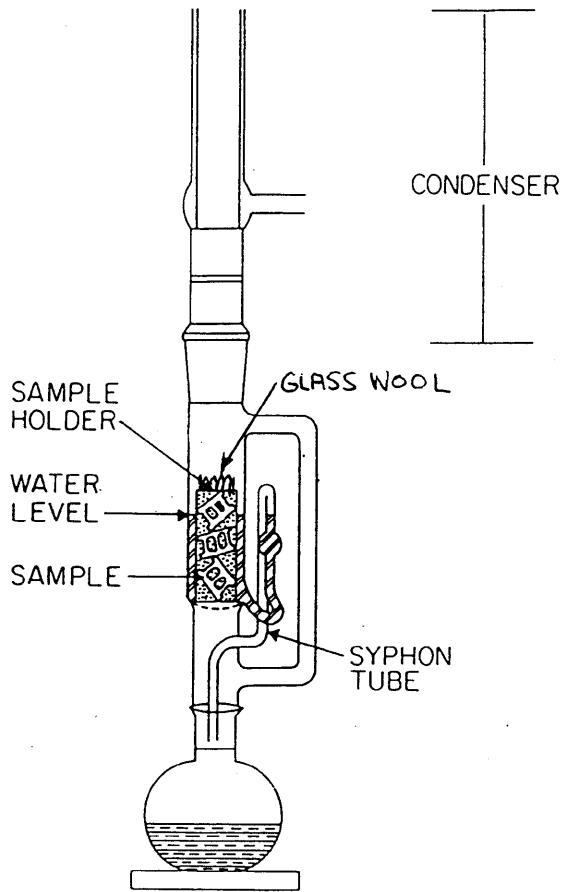


Fig. A1—Soxhlet extraction apparatus

## 1—Scope

1.1—This test method provides procedures for the sampling and analysis of hydraulic-cement mortar, concrete, or aggregate for chloride that is water-soluble and available for the corrosion reaction under the conditions of the test.

1.2—This test method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

## 2—Significance and Use

2.1—Water-soluble chloride, when present in sufficient amounts, may initiate or accelerate the corrosion of metals such as steel embedded in or contacting a cement system such as mortar, grout, or concrete. Other test methods exist for the determination of water-soluble chloride in a cement system.\* However, some aggregates contain a considerable amount of chloride that is bound in the aggregate and is not available for the corrosion reaction. The test method described in ASTM C 1218 measures a portion of the chloride contained in these aggregates. However, the amount of chloride

measured is very dependent on the degree of fineness to which the aggregates are ground during sample preparation.† The problem with the ASTM C 1218 test method is therefore twofold: the test measures chlorides that are not generally available for the corrosion reaction, and the test gives widely variable results. The test method described herein should be used when chloride-bearing aggregates influence the results obtained using ASTM C 1218.

2.2—Sulfides are known to interfere with the determination of chloride content. Blast-furnace slag aggregates and cement contain sulfide sulfur in concentrations high enough to cause significant interference and produce erroneous test results. Treatment with hydrogen peroxide, as discussed in ASTM C 114,‡ shall be used to eliminate such interference.

## 3—Apparatus

3.1—*Sampling equipment:* The apparatus required for obtaining samples by coring or sawing is described in ASTM C 42.§ Sampling by drilling is not applicable for this test and shall not be used.

3.2—*Sample processing apparatus:*

3.2.1 Samples too large to fit in the sample holder of the Soxhlet shall be reduced in size by means of a jaw crusher or by hammering.

3.2.2 Extract chlorides from the sample using a Soxhlet extractor, a schematic of which is shown in Fig. A1. The Soxhlet extractor consists of a heater, a lower flask to hold water, the sample compartment, and a condenser. The extractor contains approximately 100 ml of distilled water in the lower flask. Heat is applied to this flask; vapor from the boiling water passes to the condenser; and the condensate collects in the sample compartment. The sample is contained in a porous holder and the hot condensate collects around the sample. When the condensate reaches a critical height, the liquid is siphoned back into the lower flask and the process repeats. The nonvolatile components extracted from the sample accumulate in the lower flask, while each extraction involves fresh hot distillate. The heat input shall be sufficient to give an extraction cycle about every 20 min. For convenience, suitable commercial equipment is available.\*\*

## 4—Reagents

4.1—The reagents required for the chloride determination are given in the test method for chloride of ASTM C 114.

## 5—Sampling

5.1—Reduce the size of a minimum 300 gm sample as specified in Section 6, and divide this sample to a minimum 30 gm representative sample for use in the chloride

†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*, V. 15, No. 5, Pergamon Press, New York, Sept. 1985, pp. 863-870.

‡ASTM Test Method C 114, "Test Methods for Chemical Analysis of Hydraulic Cement."

§ASTM C 42, "Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete."

\*\*Suitable Soxhlet extraction equipment is available from Fisher Scientific (Catalog No. 09-551A) and other manufacturers.

\*ASTM Standard Test Method C 1218-92, Standard Test Method for Water-Soluble Chloride in Mortar and Concrete.

determination. If the sample is taken from concrete or mortar then the concrete or mortar shall be at least 7 days old before sampling.

Note 1—Concrete cores taken in accordance with ASTM C 42 or concrete cylinders cast from the proposed mix may be cut longitudinally or laterally to provide the required 300 gm sample representative of the core or cylinder. Experience has shown that the cooling water from core cutting will not dissolve a significant amount of chloride.

## 6— Sample Preparation

6.1—Using the jaw crusher or hammer, reduce the sample so that it fits the sample holder using the minimum crushing necessary. The sample shall not be crushed to a powder since this would release chloride bound in some aggregates which, as previously discussed, are known to not contribute to corrosion.

## 7—Procedure

7.1—A single test shall consist of determination of chloride contents of three individual 30 gm samples.

7.2—Weigh each sample ( $30 \text{ g} \pm 5 \text{ g}$ ) to the nearest 0.01 g and place in the porous sample holder of a Soxhlet extractor. Add a wad of glass wool. Place approximately 100 ml of de-ionized water in the lower flask.

7.2.1 Assemble the condenser complete with cooling water supply pipes to the extractor and place on the heater. Turn on both the heater and condenser cooling water and allow extraction to continue for 24 hr; adjust the heating rate to give a cycle about every 20 min.

7.2.2 At the conclusion of the extraction stage, transfer the solution to a 500 ml volumetric flask. Rinse the Soxhlet flask three times with distilled water, transferring the washings to the 500 ml volumetric flask; add distilled water to produce a volume of 500 ml. With a pipette transfer a 25 ml aliquot to

a 250 ml conical flask. Add 3 drops of methyl orange indicator (prepared in accordance with ASTM C 114) and add dilute (1+1) nitric acid until the solution is acidified. Add  $3.0 \pm 0.1$  ml of hydrogen peroxide (30 percent solution) to the solution. Proceed in accordance with the reference ASTM C 114, starting with the procedure specified in Section 19.5.3 and continuing to the end of Section 19.5.8.

7.2.3 Make a blank determination by using the Soxhlet, complete with thimble and glass wool, but containing no sample of cementitious material.

## 8—Calculation

8.1—Calculate percent of chloride to the nearest 0.001 percent as the average chloride content of the triplicate samples, each calculated as follows

$$\text{Chloride, percent} = 3.5453(V_1 - V_b) \times \frac{N}{M} \times \frac{500}{V_2} \quad (1)$$

where

$V_1$  = ml of 0.05 N  $\text{AgNO}_3$  solution used for titration of the sample (equivalence point)

$V_b$  = ml of 0.05 N  $\text{AgNO}_3$  solution used for titration of the blank (equivalence point)

$N$  = normality of 0.05 N  $\text{AgNO}_3$  solution, calculated to  $\pm 0.001$

$M$  = mass of concrete or mortar sample, g

$V_2$  = volume of the 25 ml aliquot determined to  $\pm 0.1$  ml (larger or smaller aliquots may be used depending on the chloride concentrations present)

8.2—Sufficient data are not available at this time to provide precision and bias statements.