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Standard Test Methods for Chemical Analysis of Thermal Insulation Materials for Leachable Chloride, Fluoride, Silicate, and Sodium Ions¹

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1. Scope

1.1 These test methods cover laboratory procedures for the determination of water-leachable chloride, fluoride, silicate, and sodium ions in thermal insulation materials in the parts per million range.

1.2 Selection of one of the test methods listed for each of the ionic determinations required shall be made on the basis of laboratory capability and availability of the required equipment and appropriateness to the concentration of the ion in the extraction solution.

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1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.4 *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

C 168 [Terminology Relating to Thermal Insulation](#)

C 692 [Test Method for Evaluating the Influence of Thermal Insulations on External Stress Corrosion Cracking Tendency of Austenitic Stainless Steel](#)

C 795 [Specification for Thermal Insulation for Use in Contact with Austenitic Stainless Steel](#)

C 871 [Test Methods for Chemical Analysis of Thermal Insulation Materials for Leachable Chloride, Fluoride, Silicate, and Sodium Ions](#)

D 1428 [Test Method for Sodium and Potassium in Water and Water-Formed Deposits by Flame Photometry](#)³

2.2 *AWWA Standards:*

4500-Si D [Molybdosilicate Method for Silica](#)⁴

4500-Si E [Heteropoly Blue Method for Silica](#)⁴

3. Terminology

3.1 *Definitions*—Refer to Terminology C 168 for definitions relating to insulation.

4. Summary of Test Methods

34.1 Insulation specimens are leached for 30 min in boiling water. Tests to determine quantitatively chloride, fluoride, silicate, and sodium ions are performed on aliquots of the filtered leachate solution.

~~3.24.2~~ *Analysis for Chloride:*

34.2.1 Amperometric-coulometric titration test method.

34.2.2 Titrimetric test method. This method is no longer recommended as requested by ASTM International due to use of a specific hazardous substance.

34.2.3 Specific ion electrode test method.

¹ These test methods are under the jurisdiction of ASTM Committee C16 on Thermal Insulation and are the direct responsibility of Subcommittee C16.31 on Chemical and Physical Properties.

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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 Withdrawn.

⁴ *Standard Methods for the Examination of Water and Wastewater*, 17th Edition, American Public Health Association, Washington, DC, 1989.

3.34.3 Analysis for Fluoride:

- 34.3.1 Specific ion electrode test method.
- 34.3.2 SPADNS colorimetric test method.

3.44.4 Analysis for Silicate:

- 34.4.1 Atomic absorption spectrophotometry test method.
- 34.4.2 Colorimetric test methods—AWWA Methods 4500-Si D and 4500-Si E.

4.5 Analysis for Sodium:

- 34.5.1 Flame photometric test method
Test Methods D 1428.
- 3.5.2 Atomic absorption spectrophotometry test method.
- 3.5.3 Sodium Ion-Selective electrode.

4.

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- 4.5.3 Sodium Ion-Selective electrode.

5. Significance and Use

4.1 It has been demonstrated that chlorides, when deposited and concentrated on the surface of austenitic stainless steel, can contribute to external stress corrosion cracking (ESCC):

5.1 Research has demonstrated that in addition to the halide ion chloride; fluoride ions, when deposited and concentrated on the surface of austenitic stainless steel, can contribute to external stress corrosion cracking (ESCC) in the absence of inhibiting ions.⁵ Analysis for fluoride has been covered because Test Methods C871 is the “source document” for other standards that require testing for leachable fluoride ions.

4.2 Testing has shown that, using the methodology of Test Method C692, neither fluoride nor iodide nor bromide initiates ESCC in the manner that can be demonstrated with chloride. After being exposed to 1500 mg/kg fluoride for 60 days with no cracking, a change to 1500 mg/kg chloride resulted in cracking in 3 days, as required by the metal qualification procedure in Test Method C692. Similar tests with iodide and bromide showed that these ions do not promote ESCC as does chloride.

4.3 Chlorides (and fluorides) can be constituents of the insulating material or of the environment, or both. Moisture in the insulation or from the environment can cause chlorides (and fluorides) to migrate through the insulation and concentrate at the hot stainless steel surface.

4.4 The presence of sodium and silicate ions in the insulation has been found to inhibit external stress corrosion cracking caused by chloride (and fluoride) ions, whether such ions come from the insulation itself or from external sources. Furthermore, if the ratio of sodium and silicate ions to chloride (and fluoride) ions is in a certain proportion in the insulation, external stress corrosion cracking as a result of the presence of chloride (and fluoride) in the insulation will be prevented or at least mitigated (see also Specification C795). Two widely used insulation specifications that are specific to ESCC allow the use of the same Test Methods C 692 and C 871 for evaluation of insulation materials. Both specifications require fluoride ions to be included with chloride ions when evaluating the extractable ions.

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5.

6. Reagents

5.1

6.1 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 Society, where

⁴ Dana, A. W., Jr., “Stress-Corrosion Cracking of Insulated Austenitic Stainless Steel,” *ASTM Bulletin No. 225*, October 1957, pp. 46–52.

⁵ Whorlow, Kenneth M., Woolridge, Edward and Hutto, Francis B., Jr., “Effect of Halogens and Inhibitors on the External Stress Corrosion Cracking of Type 304 Austenitic Stainless Steel”; STP 1320 *Insulation Materials: Testing and Applications*, Third Volume, Ronald S. Graves and Robert R. Zarr, editors, ASTM West Conshohocken, PA, 1997 page 485

such specifications are available.⁶ Use other grades only if it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5-2

6.2 *Purity of Water*— Distilled or deionized water (DI), having maximum conductivity of 2.5 $\mu\text{S}/\text{cm}$ and containing less than 0.1 ppm of chloride ions shall be used in all tests.

6. Sampling

6.1 With low-chloride insulating materials, wear clean polyethylene gloves while taking and handling the sample to avoid chloride contamination from perspiration. Do not use gloves made from chloride-containing compounds such as neoprene or saran, or materials with metallic chlorides in their formulations. Prior to use, rinse gloves twice, drain, and air-dry in a clean, halide-free environment. Store clean gloves in a closed container or envelope.

6.2 It is suitable to handle materials with more than 25 ppm chloride with clean, dry hands with no significant contamination.

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8. Test Specimen

8.1 Apparatus and tools used for special preparation and leaching shall be clean and free of chlorides, fluorides, silicates, sodium, and acidic or alkaline materials that might affect the chemical test. Distilled water must be used in all tests unless deionized water has been shown to be adequate.

8.1.1 For molded insulation, use a band saw or equivalent, making several cuts through the entire cross section of each piece of insulation to be tested. Each specimen shall be representative of the entire cross section of the piece, except that metal screen, or expanded metal used as a supportive facing shall not be included. It is recommended that thin wafers of material be cut between $\frac{1}{16}$ and $\frac{1}{8}$ in. (1.6 and 3.2 mm) thick. Cut enough material for two 20-g samples.

8.1.2 Blanket fibrous materials are cut into strips across the entire width of the blanket using clean, dry scissors.

8.1.3 Samples containing moisture are placed in a suitable container, protected from contamination, and oven dried at $230 \pm 10^\circ\text{F}$ ($100 \pm 5^\circ\text{C}$) (or manufacturers recommended temperature) to a constant weight (± 0.1 g) or overnight.

8.9. Extraction Technique

8-1

9.1 *Apparatus:*

8-1-1

9.1.1 *Electronic Balance*, capable of weighing to 2000 g with readability to the nearest 0.1 g.

8-1-2

9.1.2 *Blender*, with jar-top thread preferred.

8-1-3

9.1.3 *Beaker*, 1-L stainless or borosilicate.

8-1-4

9.1.4 *Filter*, Buchner with suitable filter paper.

8-2 Using 9.2 Using a closed-top blender, such as a 1-qt Mason jar with blender blades, blend exactly 20.0 g of sample (or other weight if necessary) in approximately 400 mL of DI or distilled water for 30 s. While most materials blend to a homogeneous mixture in 30 s, some very hard materials require 60 s or more.

8-3 Quantitatively transfer the mixture to a tared 1-L stainless steel or borosilicate beaker, rinsing with distilled or DI water.

8-4 Bring 9.4 Bring to boiling and maintain at the boiling point for 30 ± 5 min.

8-5 Remove from heat, and cool in a cold water bath to ambient temperature.

8-6 Remove water from the outside of the beaker and place on a balance. Add DI (or distilled) water to bring amount of water up to exactly 500.0 mL (g) (or other weight if necessary).

8-7 Stir 9.7 Stir mixture until it is uniform and filter through filter paper to get a clear filtrate. If not clear after the first filtration, refilter through a finer filter paper. The first small portion of filtrate is used to rinse the receiving flask and Solution A bottle. Complete this filtration by putting this filtrate in the bottle labeled Solution A. Since the relationship between solids and liquid has been established, it is not necessary to filter all of the extract. **DO NOT WASH THE FILTER CAKE!**

⁶ *Insulation Materials, Testing, and Applications, ASTM-STP-1030*, ASTM, 1990, pp. 688–698; Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards 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.

89.8 Calculate the Gravimetric Conversion Factor (GCF) by dividing the weight of the water by the weight of the sample. In the ideal case, this is $500/20 = 25$. If weights are not exactly as prescribed, a correct GCF must be calculated and used.

89.9 With calcium silicate insulation it has been shown that it is not necessary to pulverize the thin chips called for in 7.1.4.8.1.1. Equivalent results are obtained, and a lengthy filtration step is avoided, by extracting the unpulverized chips.

9.

10. Test Procedures

9.1

10.1 *Chloride Determination*—One of the following test methods shall be used on a fresh aliquot from Solution A. The precision of the test equipment is often improved through the use of analytical techniques involving known addition (or sample and standard spiking) when the ion concentrations are very low. It is recommended for chloride ion concentrations less than 20 ppm.

9.1.1

10.1.1 *Amperometric-Coulometric Titration Test Method*—

Use an apparatus⁷ in which direct current between a pair of silver electrodes causes electrochemical oxidation of the anode and produces silver ions at a constant rate. When all of the chloride ions have combined with silver ions, the appearance of free silver ions causes an abrupt increase in current between a pair of indicator electrodes. Because silver ions are generated at a constant rate, the amount used to precipitate the chloride ions is proportional to the elapsed time. Hence, the chloride content of the titration solution can be determined. Since the coulometric titrator would not discriminate between chloride, bromide, and iodide—all would test as chloride—in some cases it is practical to differentiate between the halides to show chloride only, since the others have not been shown to cause stress corrosion cracking in austenitic stainless steel. A chloride-sensitive electrode detects chloride only.

9.1.2

10.1.2 *Titrimetric Test Method*⁸—This method is no longer recommended as requested by ASTM International due to use of specific hazardous substance.

9.1.3

10.1.3 *Specific Ion Electrode Test Method*—The chloride-sensitive electrode consists of silver halide/silver sulfide membranes bonded into the tip of an epoxy electrode body. When the membrane is in contact with a chloride solution, silver ions dissolve from the membrane surface and the electrode develops a potential due to the silver ion concentration. This concentration is in turn determined by the sample chloride ion concentration. This potential is measured against a constant reference potential with a digital pH/mV meter or specific ion meter. Operation and use should follow manufacturer's recommended procedures, especially noting any corrections for interferences to determinations. The chloride-sensitive electrode is not reliable for chloride levels below 2 ppm in Solution A.

9.1.4

10.1.4 *Ion Chromatography*—It is suitable to use an ion chromatograph, following the manufacturers directions and appropriate techniques for the concentration of the ion in the extraction solution.

9.2

10.2 *Fluoride Determination*—One of the following test methods shall be used on a fresh aliquot from Solution A:

9.2.1

10.2.1 *Specific Ion Electrode Test Method for Fluoride*—The fluoride-sensitive electrode consists of a single-crystal lanthanum fluoride membrane, and an internal reference, bonded into an epoxy body. The crystal is an ionic conductor in which fluoride ions are mobile. When the membrane is in contact with a fluoride solution, an electrode potential develops across the membrane. This potential, which depends on the level of free fluoride ions in solution, is measured against an external constant reference potential with a digital pH/mV meter or specific ion meter. Operation and use should follow manufacturer's recommended procedures, especially noting any corrections for interferences to determinations.

9.2.2

10.2.2 *SPADNS Colorimetric Test Method*—This colorimetric test method is based on the reaction between fluoride and a zirconium-dye lake. The fluoride reacts with the dye lake, dissociating a portion of it into a colorless complex anion (ZrF_6^{2-}) and the dye. As the amount of fluoride is increased, the color produced becomes progressively lighter or different in hue, depending on the reagent used.

9.2.3

⁷ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards 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 Bowman, R. L., Cotlove, E., Trantham, H. V., "An Instrument and Method for Automatic, Rapid, Accurate, and Sensitive Titration of Chloride in Biologic Samples," *Journal of Laboratory and Clinical Medicine* Vol 51, 1958, pp. 461-468.

⁸ Bowman, R. L., Cotlove, E., Trantham, H. V., "An Instrument and Method for Automatic, Rapid, Accurate, and Sensitive Titration of Chloride in Biologic Samples," *Journal of Laboratory and Clinical Medicine*, Vol 51, 1958, pp. 461-468.

⁸ Clarke, F. E., "Determination of Chloride in Water Improved Colorimetric and Titrimetric Methods," *Analytical Chemistry*, Vol 22, 1950, pp. 553-555.