



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.

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

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.

and Water-Formed Deposits by Flame Photometry³

2.2 AWWA Standards:

- 4500-Si D Molybdosilicate Method for Silica⁴
- 4500-Si E Heteropoly Blue Method for Silica⁴

3. Terminology

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

4. Summary of Test Methods

4.1 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.

4.2 Analysis for Chloride:

- 4.2.1 Amperometric-coulometric titration test method.
- 4.2.2 Titrimetric test method. This method is no longer recommended as requested by ASTM International due to use of a specific hazardous substance.

4.2.3 Specific ion electrode test method.

4.3 Analysis for Fluoride:

- 4.3.1 Specific ion electrode test method.
- 4.3.2 SPADNS colorimetric test method.

4.4 Analysis for Silicate:

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

4.5 Analysis for Sodium:

- 4.5.1 Flame photometric test method Test Methods D 1428.
- 4.5.2 Atomic absorption spectrophotometry test method.
- 4.5.3 Sodium Ion-Selective electrode.

5. Significance and Use

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

³ Withdrawn.

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

external stress corrosion cracking (ESCC) in the absence of inhibiting ions.⁵ 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.

5.2 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.

5.3 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 C 795).

6. Reagents

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 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.

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.

7. Sampling

7.1 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.

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

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

⁶ *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.

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.

9. Extraction Technique

9.1 *Apparatus*:

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

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

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

9.1.4 *Filter*, Buchner with suitable filter paper.

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.

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

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

9.5 Remove from heat, and cool in a cold water bath to ambient temperature.

9.6 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).

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!**

9.8 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.

9.9 With calcium silicate insulation it has been shown that it is not necessary to pulverize the thin chips called for in 8.1.1.

Equivalent results are obtained, and a lengthy filtration step is avoided, by extracting the unpulverized chips.

10. Test Procedures

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.

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.

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.

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.

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.

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

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.

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.

10.2.3 *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.

10.3 *Silicate Determination*—One of the following test methods shall be used on a fresh aliquot from Solution A. If Solution A is cloudy, it shall be refiltered through a 0.45- μ m millipore filter or centrifuged until clear before use.

10.3.1 *Atomic Absorption Spectrophotometry Test Method*—Atomize an aliquot from Solution A by means of a nitrous oxide-acetylene flame. The absorption by the silicon atoms of radiation being emitted by a silicon hollow cathode lamp source provides a measure of the amount of silicon present in the solution, using an atomic absorption spectrophotometer.

10.3.2 *Colorimetric Test Method*—This test method covers the determination of soluble silica (SiO_2) by the molybdosilicate colorimetric procedure. In this test method, ammonium molybdate at low pH reacts with soluble silicate or phosphate to produce heteropoly acids. Oxalic acid is used to destroy the molybdophosphoric acid but not the molybdosilicic acid. The intensity of the yellow molybdosilicate complex follows Beers law. This test method is an adaption of AWWA Methods 4500-Si D and 4500-Si E. If phosphates are not present as contaminants, the oxalic acid may be omitted to obtain a more stable molybdosilicate complex.

10.3.2.1 Reagents:

(1) *10 % Ammonium Molybdate*—Dissolve 10.0 g of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in distilled water, bringing final volume to 100.0 mL.

(2) *Hydrochloric Acid*—Dilute 125 mL of concentrated HCl to 500 mL to make 1:3.

(3) *Oxalic Acid*—Dissolve 7.5 g of $H_2C_2O_4 \cdot 2H_2O$ in distilled water to make 100.0 mL.

(4) *Silica*—Prepare a standard silica solution from pure sodium metasilicate or equivalent concentrate to a stock concentration of 1000 mg/L (μ g/mL) on SiO_2 basis.

10.3.2.2 Apparatus:

(1) *Spectrophotometer*, with a 1-cm cell or tube.

(2) *Volumetric Flasks*, 50 mL, for solution and sample preparation.

(3) *Pipettes*, miscellaneous.

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

10.3.2.3 *Calibration Procedure*—Turn on the spectrophotometer and set to 410 nm. Prepare 100- $\mu\text{g}/\text{mL}$ standard by diluting 100 mL of the 1000- $\mu\text{g}/\text{mL}$ stock to 1000 mL. Pipette 1, 2, and 4 mL of the 100- $\mu\text{g}/\text{mL}$ standard into each of the three 50-mL volumetric flasks. Set out one more 50-mL flask for reagent blank use. Pipette 2.0 mL of ammonium molybdate solution into each volumetric flask. Pipette 2.0 mL of 1:3 hydrochloric acid into each flask, then bring volumes to exactly 50.0 mL, and mix well (see [Note 1](#)). Start the timer. Zero the spectrophotometer with the reagent blank. Read all three standards versus the reagent zero between 10 and 30 min of the time when the reagents were added. Plot the optical density versus millilitres of 100- $\mu\text{g}/\text{mL}$ standard added ($\mu\text{g}/\text{mL}$ in test solution).

NOTE 1—When it has been determined that phosphates are present, 2.0 mL of oxalic acid must be added after the other two reagents. When oxalic acid is used, the timing is much more important, because the yellow color begins to fade after 15 min.

10.3.2.4 *Determination of the Unknown*—Test as soon as possible after cooling Solution A, preferably on the same day, overnight at worst. In a 50-mL volumetric flask, add 1.0 mL of Solution A (see [Note 2](#)), 2.0 mL of ammonium molybdate reagent, and 2.0 mL of 1:3 hydrochloric acid, (and 2 mL oxalic acid solution when necessary), followed by swirling to dissolve any precipitated material. Add distilled (or DI) water to bring volumes to exactly 50.0 mL. Mix well and start the timer. Between 10 and 30 min, read the sample versus the reagent zero. Consult the calibration curve to find equivalent millilitres of 100- $\mu\text{g}/\text{mL}$ standard.

NOTE 2—This test method was designed for determining soluble silicate in materials containing 1000 to 10 000 ppm of soluble silicate as determined by this test method. For materials out of this range, more or less of Solution A must be used with appropriate adjustments being made to the calculation procedure.

10.4 *Sodium Determination*—One of the following test methods shall be used on 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 sodium ion concentrations less than 500 ppm.

10.4.1 *Flame Photometric Test Method*—Atomize the filtered aliquot in a flame and determine the concentration of sodium by photometry in accordance with Test Methods [D 1428](#) or equivalent.

NOTE 3—Corrections must be made if high concentrations of calcium, potassium, or magnesium are present.

10.4.2 *Atomic Absorption Spectrophotometry Test Method*—Atomize the filtered aliquot by means of an air-acetylene flame. The absorption by the sodium atoms of radiation being emitted by a sodium hollow cathode lamp source provides a measure of the amount of sodium present in the solution, using an atomic absorption spectrophotometer.

NOTE 4—Corrections must be made if high concentrations of calcium, potassium, or magnesium are present.

10.4.3 *Ion-Selective Electrode*—Use the Sodium Ion-Selective electrode according to the manufacturer's directions, calibrating with standards to bracket the range of the unknown.

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

10.5 *pH, when required*—Determine the pH^8 of an aliquot from Solution A. Discard the aliquot after the determination. The pH shall be run as quickly as possible after the extraction because solutions sometimes change pH on standing.

11. Calculation

11.1 *Procedure*—The gravimetric conversion factor (GCF) is calculated using Eq 1:

$$GCF = \{\text{Volume liquid (g)}\} / \{\text{Sample weight (g)}\} \quad (1)$$

11.2 Chloride content of insulation is calculated using Eq 2:

$$\text{Cl}^- (\mu\text{g/g}) = \text{concentration observed in Solution A } (\mu\text{g/mL}) \times GCF \quad (2)$$

11.3 Fluoride content of insulation is calculated using Eq 3:

$$\text{F}^- (\mu\text{g/g}) = \text{concentration observed in Solution A } (\mu\text{g/mL}) \times GCF \quad (3)$$

11.4 Silicate content of insulation is calculated using Eq 4:

$$\text{SiO}_3 = (\mu\text{g/g}) = \text{concentration of SiO}_2 \text{ observed in Solution A } (\mu\text{g/mL}) \times 76/60 \times GCF \quad (4)$$

NOTE 5—The factor 76/60 converts observed SiO_2 to $\text{SiO}_3 =$ as required by Specification [C 795](#) and other specifications relating to austenitic stainless steel corrosion.

11.5 Sodium content of insulation is calculated using Eq 5:

$$\text{Na}^+ (\mu\text{g/g}) = \text{concentration observed in Solution A } (\mu\text{g/mL}) \times GCF \quad (5)$$

12. Report

12.1 Include in the report of the results of each test the following information:

12.1.1 Any pertinent information concerning the identification of the material.

12.1.2 The test methods used for determination of chloride, fluoride, sodium, and silicate.

12.1.3 The numerical results of the tests expressed in $\mu\text{g/g}$ (ppm) of chloride, fluoride, sodium, and silicate calculated on the basis of weight of each dry insulation specimen.

13. Precision and Bias

13.1 A round robin was conducted utilizing three different types of insulation and two prepared extracts. Samples were supplied to ten laboratories. Seven reported results on all of the samples, and one reported partial results.

13.1.1 Of the 236 data points supplied, 39 were thrown out as outliers because they were either double or one half of the average of the other comparative data points. Twenty-six of the

TABLE 1 Round-Robin Mean Values and Standard Deviations

Sample Type	Calsil	Perlite	Fiber Glass	Solution	Solution
Chloride mean	121	40.3	116	129	54.2
Standard deviation	13.4	8.9	22.5	16.3	12.6
sd/mean × 100	11.1	22.1	19.3	12.6	23.2
Fluoride mean	55.6	17.5	1.9	44.0	13.2
Standard deviation	7.4	3.7	0.8	2.9	2.8
sd/mean × 100	13.3	21.1	42.1	6.6	21.2
Sodium mean	5845	23 950	732	5243	1692
Standard deviation	1472	3531	194	595	523
sd/mean × 100	25.1	14.7	26.5	11.3	30.9
Silicate mean	5926	45 860	498	4275	1358
Standard deviation	1609	5190	206	831	295
sd/mean × 100	27.2	11.3	41.3	19.4	21.7

outliers were from three laboratories that were using the ion chromatograph. Of these 26, 6 and an additional 4 were from a laboratory that used an unapproved extraction method.

13.1.2 For each determination, a mean value and a standard deviation were calculated. **Table 1** shows the results.

13.1.3 There was no estimation of bias, since the true values for the samples were unknown.

13.1.4 Since the material samples were run in duplicate by all of the participating laboratories but one, a within laboratory value could have been calculated but was not. Suffice it to say that the within laboratory variability should have been somewhat less than the between laboratory variability.

13.1.5 A full report of the data is on file with ASTM as a research report.⁹

13.2 An interlaboratory round-robin¹⁰ was performed by five laboratories on samples taken from large batches of prepared extraction solutions from six different kinds of thermal insulation containing a wide range of extractable ions concentrations. Each lab ran the test series three times provid-

ing interlaboratory and within-laboratory precision data. The results of these tests are for the chemical analysis only and do not include the extraction step, which was covered by the previous precision and bias testing.

13.2.1 The interlaboratory reproducibility standard deviation as a percentage of the mean value obtained by all of the labs (SD/Mean x 100) was higher at the low ion concentrations and therefore the data is summarized with ranges:

Silicate <1000ppm = 12.1%; >1000ppm = 7.5%
 Sodium <600ppm = 21.7%; >600ppm = 9.0%
 Chloride <20ppm = 23.6%; >20ppm = 10.0%
 Fluoride <5ppm = 34.1%; >5<10ppm = 13.4%; >10ppm = 5.7%

13.2.2 The within-laboratory repeatability standard deviation as a percentage of the mean value obtained within the lab (SD/Mean x 100) is summarized as follows:

Silicate <1000ppm = 5.4%; >1000ppm = 2.6%
 Sodium <600ppm = 5.7%; >600ppm = 1.8%
 Chloride <20ppm = 9.6%; >20ppm = 7.4%
 Fluoride <5ppm = 4.0%; >5<10ppm = 2.5%; >10ppm = 3.0%

14. Keywords

14.1 chemical analysis; chloride; fluoride; silicate; sodium; thermal insulation

⁹ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: C16-1015.

¹⁰ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: C16-1029.

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