Designation: C 871 – 95

Standard Test Methods for Chemical Analysis of Thermal Insulation Materials for Leachable Chloride, Fluoride, Silicate, and Sodium Ions[1]

This standard is issued under the fixed designation C 871; 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 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 should be made on the basis of laboratory capability and availability of the required equipment.

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 692 Test Method for Evaluating the Influence of Thermal Insulations on the External Stress Corrosion Cracking Tendency of Austenitic Steel[2]

C 795 Specification for Thermal Insulation for Use in Contact with Austenitic Stainless Steel[2]

D 1428 Test Methods 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. Summary of Test Methods

3.1 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.

3.2 *Analysis for Chloride*:

3.2.1 Amperometric-coulometric titration test method.

3.2.2 Titrimetric test method.

3.2.3 Specific ion electrode test method.

3.3 *Analysis for Fluoride*:

3.3.1 Specific ion electrode test method.

3.3.2 SPADNS colorimetric test method.

3.4 *Analysis for Silicate*:

3.4.1 Atomic absorption spectrophotometry test method.

3.4.2 Colorimetric test methods—AWWA Methods 4500-Si D and 4500-Si E.

3.5 *Analysis for Sodium*:

3.5.1 Flame photometric test method
Test Methods D 1428.

3.5.2 Atomic absorption spectrophotometry test method.

3.5.3 Ross Sodium Ion-Sensitive electrode.[5]

4. 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).[6] Analysis for fluoride has been covered because Test Methods C 871 is the "source document" for other standards that require testing for leachable fluoride ions.

4.2 Testing[7] has shown that, using the methodology of Test Method C 692, 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 C 692. Similar tests with iodide and bromide showed that these ions do not promote ESCC as does chloride.

4.3 Chlorides (and fluorides) may 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

---

[1] These test methods are under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and are the direct responsibility of Subcommittee C16.31 on Chemical and Physical Properties.

Current edition approved June 15, 1995. Published August 1995. Originally published as C 871 – 77. Last previous edition C 871 – 94.

[2] *Annual Book of ASTM Standards*, Vol 04.06.

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

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

[5] Available from VWR Scientific, Box 39396, Denver, CO 80239.

[6] Dana, A. W., Jr., "Stress-Corrosion Cracking of Insulated Austenitic Stainless Steel," *ASTM Bulletin No. 225*, October 1957, pp. 46–52.

[7] *Insulation Materials, Testing, and Applications, ASTM STP 1030,* ASTM, 1990, pp. 688–698.

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).

5. Reagents

5.1 *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.[8] Other grades may be used, provided 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 *Purity of Water*—Distilled or deionized water (DI), having maximum conductivity of 2.5 µS/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 Materials with more than 25 ppm chloride may be handled with clean, dry hands with no significant contamination.

7. Test Specimen

7.1 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.

7.1.1 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 1⁄16 and 1⁄8 in. (1.6 and 3.2 mm) thick. Cut enough material for two 20-g samples.

7.1.2 Blanket fibrous materials may be cut into strips across the entire width of the blanket using clean, dry scissors.

7.1.3 Each sample should be placed in a suitable container, protected from contamination, and oven dried at 230 ± 10°F (100± 5°C) to a constant weight (±0.1 g) or overnight.

8. Extraction Technique

8.1 *Apparatus*:

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

8.1.2 *Blender*, Osterizer[9] with jar-top thread preferred.

8.1.3 *Beaker*, 1-L stainless or Pyrex.

8.1.4 *Filter*, Buchner with suitable filter paper.[10]

8.2 Using a closed-top blender, such as a 1-qt Mason jar with Osterizer blender blades, blend exactly 20.0 g of sample 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 may require 60 s or more.

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

8.4 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).

8.7 Stir mixture until it is uniform and filter through filter paper[10] to get a clear filtrate. If not clear after the first filtration, refilter through a finer filter paper. The first 100 to 200 mL of filtrate should be 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!

8.8 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.

8.9 With calsil it has been shown that it is not necessary to pulverize the thin chips called for in 7.1.1. Equivalent results are obtained, and a lengthy filtration step is avoided, by extracting the unpulverized chips.

9. Test Procedures

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

9.1.1 *Amperometric-Coulometric Titration Test Method*—Use an apparatus[11] 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

---

[8] *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.

[9] One such apparatus found acceptable is the 10-speed Osterizer, manufactured by the Oster Division, Sunbeam Corporation in Milwaukee, WI 53217. While Oster manufactures several models, all use the “jar-top thread” on the blade assembly, making it possible to use a 1-qt Mason jar for the pulverization step of the procedure.

[10] Whatman 41, GF-A, or other filter paper is suitable for this purpose and commercially available.

[11] 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. An apparatus found acceptable is the Aminco-Cotlove Chloride Titrator, manufactured by American Instrument Co., Silver Springs, MD 20907.

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 may be necessary 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 *Titrimetric Test Method*[12]—Add dilute mercuric nitrate solution to an acidified aliquot in the presence of diphenylcarbazone indicator. At the mercury-chloride equivalence point, a blue-violet, mercury-diphenylcarbazone complex forms, which is proportional in intensity to the excess of mercury ion present. The titrimetric procedure is independent of practically all common interferences.

9.1.3 *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.[13] 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.

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

9.2.1 *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 *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.

9.3 *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-µm millipore filter before use.

9.3.1 *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.

9.3.2 *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.

9.3.2.1 *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 from "Dilut-it"[5] or equivalent concentrate to a stock concentration of 1000 mg/L (µg/mL) on SiO_2 basis.

9.3.2.2 *Apparatus*:

(1) Spectrophotometer,[14] with a 1-cm cell or tube.

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

(3) Pipettes, miscellaneous.

9.3.2.3 *Calibration Procedure*—Turn on the spectrophotometer and set to 410 nm. Prepare 100-µg/mL standard by diluting 100 mL of the 1000-µg/mL stock to 1000 mL. Pipet 1, 2, and 4 mL of the 100-µg/mL standard into each of the three 50-mL volumetric flasks. Set out one more 50-mL flask for reagent blank use. Pipet 2.0 mL of ammonium molybdate solution into each volumetric flask. Pipet 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-µg/mL standard added (µg/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.

9.3.2.4 *Determination of the Unknown*—Test as soon as possible after cooling Solution A, preferably on the same day,

---

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

[13] An acceptable pH meter is the Orion pH Meter, Model 407A, Orion Research, Inc., Boston, MA 02129.

[14] An acceptable spectrophotometer is the Perkin-Elmer Atomic Absorption Spectrophotometer, Model 403, manufactured by Perkin-Elmer, Norwalk, CT 06856.

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-µg/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.

9.4 *Sodium Determination*—One of the following test methods shall be used on Solution A.

9.4.1 *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.

9.4.2 *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.

9.4.3 *Ross Ion-Sensitive Electrode*[5]—Use the Ross Sodium Ion-Sensitive electrode according to the manufacturer's directions, calibrating with standards to bracket the range of the unknown.

9.5 *pH*, when required—Determine the pH[12] of an aliquot from Solution A. Discard the aliquot after the determination. The pH should be run as quickly as possible after the extraction because solutions sometimes change pH on standing.

10. Calculation

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

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

10.2 Chloride content of insulation is calculated using Eq 2:

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

10.3 Fluoride content of insulation is calculated using Eq 3:

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

10.4 Silicate content of insulation is calculated using Eq 4:

$$SiO_3^{=}\ (\mu g/g) = \text{concentration of } SiO_2 \text{ observed in Solution A } (\mu g/mL) \times 76/60 \times GCF \quad (4)$$

NOTE 5—The factor 76/60 converts observed SiO_2 to $SiO_3^{=}$ as required by Specification C 795 and other specifications relating to austenitic stainless steel corrosion.

10.5 Sodium content of insulation is calculated using Eq 5:

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

11. Report

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

11.1.1 Any pertinent information concerning the identification of the material,

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

11.1.3 The numerical results of the tests expressed in µg/g (ppm) of chloride, fluoride, sodium, and silicate calculated on the basis of weight of each dry insulation specimen.

12. Precision and Bias

12.1 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.

12.1.1 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 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.

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

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

12.4 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.

12.5 A full report of the data is on file with ASTM as a research report.[15]

13. Keywords

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

---

[15] Supporting data are available from ASTM Headquarters. Request RR: C16–1015.

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

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

This standard is copyrighted by ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (http://www.astm.org).