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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 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 *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 692 Test Method for Evaluating the Influence of Thermal Insulations on ~~the~~ External Stress Corrosion Cracking Tendency of Austenitic Stainless Steel

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

D 1428 Test Method~~s~~ 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.

~~3.2.2Titrimetric test method.~~

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

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.

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

[3] Withdrawn.

~~[4] Available from VWR Scientific, Box 39396, Denver, CO 80239.~~

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

3.5 *Analysis for Sodium*:

3.5.1 Flame photometric test method

Test Methods D 1428.

3.5.2 Atomic absorption spectrophotometry test method.

~~3.5.3Ross Sodium Ion-Sensitive electrode.~~

3.5.3 Sodium Ion-Selective electrode.

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).[5] 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[6] 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) 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 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.[7] Use other grades only if 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 It is suitable to handle materials with more than 25 ppm chloride 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 are cut into strips across the entire width of the blanket using clean, dry scissors.

7.1.3 Samples containing moisture are placed in a suitable container, protected from contamination, and oven dried at 230 ± 10°F (100± 5°C) (or manufacturers recommended temperature) to a constant weight (±0.1 g) or overnight.

8. Extraction Technique

8.1 *Apparatus*:

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

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

[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.

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

8.1.2 *Blender*, ~~Osterizer~~ with jar-top thread preferred.

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

8.1.4 *Filter*, Buchner with suitable filter paper.

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 (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 ~~Pyrex~~borosilicate 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) (or other weight if necessary).

8.7 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!

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. 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 *Amperometric-Coulometric Titration Test Method*—
Use an apparatus[8] 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 *Titrimetric Test Method*~~—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]—This method is no longer recommended as requested by ASTM International due to use of specific hazardous substance.

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

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

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~~[8] 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.~~

[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.

~~[9] Whatman 41, GF-A, or other filter paper is suitable for this purpose and commercially available.~~

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