



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 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 Steel²

C 795 Specification for Thermal Insulation for Use in Contact with Austenitic Stainless Steel²

D 1428 Test Methods for Sodium and Potassium in Water 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. 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 Testing⁷ 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

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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.