



Designation: C 1226 – 93 (Reapproved 2006)

Standard Test Method for Soluble Chlorides in Asbestos¹

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1. Scope

1.1 This test method covers the leaching out of the soluble chlorides in asbestos and the volumetric determination of chloride ion in the leachate.

1.2 **Warning**—Breathing of asbestos dust is hazardous. Asbestos and asbestos products present demonstrated health risks for users and for those with whom they come into contact. In addition to other precautions, when working with asbestos-cement products, minimize the dust that results. For information on the safe use of chrysotile asbestos, refer to “Safe Use of Chrysotile Asbestos: A Manual on Preventive and Control Measures.”²

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. See 1.2 for a specific hazard warning.*

2. Referenced Documents

2.1 ASTM Standards:³

D 1193 Specification for Reagent Water

D 2590 Test Method for Sampling Chrysotile Asbestos

D 2946 Terminology for Asbestos and Asbestos–Cement Products

D 3879 Test Method for Sampling Amphibole Asbestos

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

2.2 QAMA Standards:

A-2-72 Definitions of Terms Relating to Asbestos⁴

G-5-74 Soluble Chlorides⁴

¹ This test method is under the jurisdiction of ASTM Committee C17 on Fiber-Reinforced Cement Products and is the direct responsibility of Subcommittee C17.03 on Asbestos-Cement Sheet Products and Accessories.

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² Available from The Asbestos Institute, http://www.chrysotile.com/en/sr_use/manual.htm.

³ 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.

⁴ Published in the *Chrysotile Asbestos Test Manual*. Available from the Asbestos Institute, 1002 Sherbrooke St. W., Suite 1750, Montreal, QC, Canada H3A 3L6.

2.3 ACS Standards:

Specifications of the Committee on Analytical Reagents⁵

3. Terminology

3.1 Refer to Terminology D 2946 and QAMA Standard A-2-72.

4. Summary of Test Method

4.1 The asbestos is leached with water in a Soxhlet extractor for 3 h.

4.2 Dilute mercuric nitrate solution is added to an acidified specimen of leachate in the presence of mixed diphenyl-carbazone bromphenol blue indicator. The end point of the titration is the formation of the blue-violet mercury diphenyl-carbazone complex (see Note 5).

4.3 An alternative titration by means of an automatic titrator is presented in Annex A1.

4.4 An alternative simplified titration, suitable for non-referee internal quality control, is presented in Annex A2.

5. Significance and Use

5.1 This test method provides an evaluation of the water-soluble chlorides in asbestos. It is used to determine the suitability of asbestos for use in products, such as gaskets, that may be in contact with metals under hydrothermal conditions that foster chloride ion corrosion.

6. Interferences

6.1 Zinc, lead, nickel, ferrous, and chromous ions affect the solubility of the chloride ion and the end point color, but they do not reduce the accuracy of the titration when present in concentrations up to 100 ppm.

6.2 Copper is tolerable up to 50 ppm.

6.3 Titration in the presence of chromate ion requires an indicator with intensified background color, such as alphasaurine, and prior reduction for concentrations above 100 ppm.

⁵ *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.

6.4 Ferric ion above 10 ppm must be reduced before titration, and sulphite ion must be oxidized.

6.5 A part of any bromide and fluoride ion that is present will also be titrated with the chloride ion.

6.6 Quaternary ammonium salts also interfere, if present in significant concentrations (1 to 2 ppm).

6.7 Deep coloration of the leachate may also interfere.

7. Apparatus

7.1 *Microburet*, 1 or 5-cm³ capacity, with 0.01-cm³ graduation intervals (see [Note 5](#)).

7.2 *Soxhlet Extraction Apparatus*, including:

7.2.1 Flask, 500 cm³, with a 24/40 glass joint, flask to tube.

7.2.2 *Extraction Tube*, with a 43-mm diameter by 123-mm length thimble, with a 55/50 glass joint, tube to condenser.

7.2.3 *Condenser*, with a 55/50 glass joint to tube.

7.2.4 *Porous Thimble Specimen Holders*.

7.3 *Desiccator*.

7.4 *Tweezers*.

7.5 Refer also to [A1.1](#).

8. Reagents and Materials (see [Note 5](#))

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All reagents shall conform to the American Chemical Society specifications. Other grades may be used, provided it is first ascertained that the reagents are of sufficiently high purity to permit their use without decreasing the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type III of Specification [D 1193](#).

8.2.1 In addition, reagent water shall be free of chloride ion.

8.3 *Bromophenol Blue*, (3', 3'', 5', 5'' tetrabromophenolsulphonphthalein), powder.

8.4 *Diphenylcarbazone* (C₆H₅NHNHCON: NC₆H₅·C₆H₅NHNHCONHNHC₆H₅), crystalline.

8.5 *Ethanol* (CH₃CH₂OH (ethyl alcohol), 95 %.

8.6 *Hydrogen Peroxide* (H₂O₂)—30 % solution.

8.7 *Hydroquinone* (C₆H₆O₂)—10 g/dm³.

8.7.1 Dissolve 1 g of purified hydroquinone in water and dilute to 100 cm³.

8.8 *Mercuric Nitrate Solution*—(Hg(NO₃)₂·H₂O), 0.025 N.

8.8.1 Acidify 50 cm³ of water with 0.5 cm³ of nitric acid, HNO₃, sp gr 1.42.

8.8.2 Dissolve 4.2830 g of Hg(NO₃)₂·H₂O in the acidified water.

8.8.3 Dilute to 1 dm³.

8.8.4 Filter, if necessary.

8.8.5 Standardize against the standard NaCl solution, using the procedure in [12.13](#).

NOTE 1—The end point, while sharp, can be improved somewhat for certain types of leachates by adding to the titration specimen several drops of a 0.05-g/cm³ solution of xylene cyanole FF or alphazurine blue-green dye (color index 714). These chemicals can be mixed with the indicator in the same proportions.

8.9 *Mercuric Nitrate Solution*—(Hg(NO₃)₂·H₂O), 0.0141 N.

8.9.1 Acidify 25 cm³ of water with 0.25 cm³ of nitric acid, HNO₃ sp gr 1.42.

8.9.2 Weigh out 2.5200 g of Hg(NO₃)₂·H₂O.

8.9.3 Dissolve this Hg(NO₃)₂·H₂O in the acidified water ([8.8.1](#)) and dilute to 1 dm³.

8.9.4 Filter, if necessary.

8.9.5 Standardize against the standard NaCl solution, using the procedure in [12.13](#) (see [Note 2](#)).

8.10 *Mixed Indicator*.

8.10.1 Dissolve 0.5 g of crystalline diphenylcarbazone and 0.05 g of bromophenol blue powder in 75 cm³ of ethanol 95 % ([Note 2](#)), and dilute to 100 cm³ with the ethanol.

NOTE 2—Denatured alcohol is not suitable. Methanol or isopropanol may be used if the 95 % ethanol is not available.

8.10.2 Store the mixed indicator in a brown bottle and discard after six months ([Note 3](#)).

NOTE 3—The indicator solution generally deteriorates to the point that it yields no end point color after 12 to 18 months. Temperatures above 38°C (100°F) and exposure to bright light may shorten its useful life. A dry powder mixture of the two indicator ingredients is stable for much longer periods. Both the powder mixture (capsule form) and the liquid indicator are available commercially.

8.11 *Nitric Acid* (HNO₃), sp gr 1.42.

8.12 *Nitric Acid Solution* (HNO₃), (3 + 997).

8.12.1 Dissolve 3 cm³ of HNO₃ sp gr 1.42 in water and dilute to 1 dm³.

8.13 *pH Indicating Paper*, long-range type, covering a pH range from 1 to 11.

8.14 *Sodium Chloride Standard Solution* (NaCl) (0.025 N).

8.14.1 Dry 1.5 g of NaCl for 1 h at 600°C.

8.14.2 Cool in a desiccator.

8.14.3 Weigh out 1.4613 ± 0.0002 g of the dried NaCl.

8.14.4 Dissolve in water and dilute with water at 20°C to 1 dm³ in a volumetric flask.

NOTE 4—Drying for 2 h at 105°C is adequate for practically all analytical requirements. If ultimate accuracy of standardization is desired, fuse the NaCl prior to cooling it in a desiccator.

8.15 *Sodium Hydroxide Solution* (NaOH), 2 g of NaOH per dm³.

8.15.1 Dissolve 2 g of NaOH in water and dilute to 1 dm³.

8.16 *Xylene Cyanole FF*—(OSO₂(NaOSO₂)(HO)C₆H₂C [C₆H₃(CH₃)NHC₂H₅], CAS)—No. 2650-17-1, or alphazurine blue green dye (color index 714).

8.17 See also [A1.2](#) and [A2.1](#).

9. Hazards

9.1 Most of the reagents in Section 8 are toxic and some are corrosive. Wear protective clothing, gloves, and goggles.

9.2 **Warning**—see [1.2](#).

10. Sampling, Test Specimens, and Test Units

10.1 *Sampling*:

10.1.1 Sample chrysotile asbestos in accordance with Test Method [D 2590](#).

10.1.2 Sample amphibole asbestos in accordance with Test Method [D 3879](#).

10.2 *Test Specimens*:

10.2.1 The 25-g test specimens shall be derived from the laboratory test sample. Wear gloves to prevent contact of the asbestos with sweat on the skin.