



Standard Test Method for Inorganic Chlorides and Sulfates in Insulating Oils¹

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This standard has been adopted for use by government agencies to replace Method 5703 of Federal Test Method Standard No. 791b.

^{ε1} NOTE—Editorial changes were made in January 2002.

1. Scope

1.1 This test method describes the qualitative determination of inorganic chlorides and sulfates in electrical insulating oils.

NOTE 1—It should be pointed out that if the strong acids, hydrochloric and sulfuric, are present, they will give positive results for chlorides and sulfates, respectively.

1.2 *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:*
D 1193 Specification for Reagent Water²

3. Summary of Test Method

3.1 Water is used to extract inorganic chlorides and sulfates from a test specimen of insulating oil. A portion of the water layer is treated with nitric acid and silver nitrate to reveal a white precipitate if chloride is present. A second portion is treated with hydrochloric acid and barium chloride. A white precipitate indicates the presence of sulfate.

4. Significance and Use

4.1 The presence of inorganic chlorides and sulfates in oil in contact with metal surfaces in electrical equipment can result in corrosion of transformer components. The deterioration of metal surfaces is dependent upon the quantity of the corrosive agent and the operating environment. Although not quantitative, this test method is a means for recognizing the potential for such a hazard.

5. Apparatus

- 5.1 *Separatory Funnel*, 250-mL and 500-mL capacity.
5.2 *Beakers*, 250-mL capacity.
5.3 *Test Tubes*, 25-mL capacity.

6. Reagents

6.1 *Purity of Reagents*—Use reagent grade chemicals 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.³ 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.

6.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D 1193, Type I or Type II.

6.3 *Barium Chloride Solution (100 g/L)*—Dissolve 100 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 L.

6.4 *n-Heptane*.

6.5 *Hydrochloric Acid (relative density 1.19)*—Concentrated hydrochloric acid (HCl).

6.6 *Nitric Acid (relative density 1.42)*—Concentrated nitric acid (HNO_3).

6.7 *Silver Nitrate Solution (100 g/L)*—Dissolve 100 g of silver nitrate (AgNO_3) in water and dilute to 1 L.

7. Procedure

7.1 In a 250-mL separatory funnel, shake thoroughly 100 mL (100 cm³) of the oil test specimen with 75 mL of water at 95°C. Allow to separate, then draw off the water layer into a 250-mL beaker.

7.1.1 In case the separated water is not clear, filter it through a 10-cm diameter qualitative filter paper into a 250-mL beaker.

¹ This test method is under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.06 on Chemical Test.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *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.

The filtrate is then used for tests. As a check on the filter paper, 75 mL of hot water, passed through the paper prior to filtering the separated water, should give negative tests for chlorides and sulfates.

7.1.2 In the case of high-viscosity polybutene, dilute the 100-mL sample with an equal amount of *n*-heptane. Stir until the test specimen is completely dissolved, transfer the solution to a 500-mL separatory funnel, and proceed in accordance with 7.1.

NOTE 2—Polybutenes with a viscosity of 7×10^{-4} m²/s at 100°C can be accommodated. Polybutenes with viscosities greater than 7×10^{-4} m²/s at 100°C have not been evaluated.

7.2 Pour 15 mL of the separated water layer into a clean test tube, add a few drops of HNO₃, and then add 5 mL of AgNO₃ solution. The appearance of a white precipitate indicates the presence of chlorides.

7.3 Pour a second portion of 15 mL of the water into a clean test tube. Heat to the boiling point, add 5 drops of HCl, and then slowly add 5 mL of BaCl₂ solution. A white precipitate reveals the presence of sulfates.

8. Report

8.1 Report the following information:

- 8.1.1 Sample identification,
- 8.1.2 The presence or the absence of chlorides and sulfates individually, and
- 8.1.3 The detection limit of ions (ppm) supported by the test method.

9. Precision and Bias

9.1 No statement is made about the precision or the bias of this test method, since the results merely state whether there is a conformance to the criteria for success specified in the procedure and is not quantitative. The detection limit of inorganic sulfates and chlorides detected by this test method has been measured by one laboratory at 6 and 20 ppm, respectively. These measurements are supported by a research report.⁴

10. Keywords

10.1 chlorides; inorganic; insulating oil; sulfates

⁴ Available from ASTM Headquarters. Request RR:D27-1009.

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