



Standard Test Method for Chlorine Content of Polybutenes Used for Electrical Insulation¹

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^{ε1} NOTE—The mercury warning was editorially added in April 2009.

^{ε2} NOTE—In 7.7, 20 L was changed to 2 L editorially in August 2009.

1. Scope

1.1 This test method describes the determination of the total chloride content of polybutenes used for electrical insulation.

1.2 **Warning**—Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website (<http://www.epa.gov/mercury/faq.htm>) for additional information. Users should be aware that selling mercury or mercury-containing products, or both, in your state may be prohibited by state law.

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.* For specific hazards information, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards*:²

[D878 Test Method for Inorganic Chlorides and Sulfates in Insulating Oils](#)

[D1193 Specification for Reagent Water](#)

3. Summary of Test Method

3.1 Organically bound chlorine is converted into sodium chloride by reaction with sodium biphenyl solution. The

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 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.

sodium chloride formed is extracted with dilute nitric acid, and the chlorine content of the aqueous phase is determined by potentiometric titration.

4. Significance and Use

4.1 Chlorine is normally present in polybutenes in small amounts, usually below 50 ppm, as organically bound chlorine. Inorganic chloride is normally not present.

NOTE 1—The qualitative presence or absence of inorganic chloride may be tested by Test Method [D878](#).

5. Interferences

5.1 The presence of substances which form insoluble silver compounds, such as sulfides, will give high results. Such substances are not normally present in polybutenes.

6. Apparatus

6.1 *Separatory Funnel*, 250-mL.

6.2 *Potentiometric Titrimeter*, automatic recording, or manual.

6.3 *Electrodes*:

6.3.1 Silver and glass electrode combination is preferred.

6.3.2 A silver electrode with a mercurous sulfate reference electrode is an acceptable alternative.

6.4 *Microburet*, 5-mL, with 0.01-mL divisions.

7. Reagents

7.1 *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,

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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean reagent grade water as defined by Type I conforming to Specifications **D1193**.

7.3 Dilute Nitric Acid (2.1 M)—Dilute 134 mL of concentrated nitric acid to 1.0 L with water.

7.4 Isopropyl Alcohol.

7.5 Silver Nitrate, Standard Solution (0.025 N)—Weigh accurately 0.4247 g of silver nitrate (AgNO_3). Transfer it to a 1-L volumetric flask and add water to dissolve. Add 3.0 mL of concentrated nitric acid (HNO_3 , relative density (specific gravity) 1.42) and then add water to the 1-L mark of the volumetric flask. Standardize this solution against a pure chloride standard. Check the solution at least monthly to assure a constant reagent.

7.6 Sodium Chloride, NIST Standard Reference Material 919A.

NOTE 2—Dry the silver nitrate overnight in a desiccator before making up the solution. Both the solid material and the solution must be protected from light by storage in brown glassware in the dark.

7.7 Sodium Biphenyl Solution—Transfer 300 mL of dry toluene and 58 g of metallic sodium to a 2 L, three-necked flask, equipped with a heating mantle, nitrogen gas inlet, mercury seal stirrer, and reflux condenser. Heat until the toluene refluxes and the sodium melts completely. Start the stirrer, and stir until the sodium is finely dispersed. Cool to less than 10°C in a suitable bath (not water). Remove the condenser, and add 1250 mL of dry ethylene glycol dimethyl ether. While stirring and passing nitrogen gas over the mixture, add 390 g of biphenyl. The reaction should start immediately, as evidenced by the green color of sodium biphenyl. The temperature of the reaction mixture should be kept below 30°C. When the reaction is complete (1½ to 2 h), pour the reagent into dry 500-mL brown prescription bottles with screw caps and foil liners. The reagent is stable for several months if refrigerated. (If any unreacted sodium remains in the reaction flask, add 100 mL of isopropyl alcohol, and place the flask in a hood until the metal has dissolved.)⁴

NOTE 3—Two vials (30 mL) of this reagent are normally required to give excess reagent.

8. Hazards

8.1 Consult OSHA regulations and suppliers' Material Safety Data Sheets for all materials used in this test method.

9. Procedure

9.1 Dissolve 35.5 ± 0.1 g of polybutene in 25 mL toluene in a 150-mL beaker by stirring with a small glass rod. Transfer

the solution to a separatory funnel. Rinse the beaker several times with a total of 25 mL toluene and add the rinses to the funnel.

9.2 Add an excess of sodium biphenyl solution into the separatory funnel. Excess reagent is evidenced by a blue or green color.⁴ (See **Note 2**.) Stopper and mix thoroughly by gentle shaking. Vent occasionally to release slight pressure.

9.3 Allow the blue-green mixture to stand 5 min to ensure complete reaction. Remove stopper, add 2 mL of isopropyl alcohol, and swirl with stopper removed until excess reagent is destroyed.

9.4 Add slowly 50 mL of dilute nitric acid. Contact organic and aqueous phases by gentle swirling and rocking for about 5 min. Loosen the stopper occasionally to release slight pressure. Drain the aqueous phase into a beaker. Extract the organic phase twice more with 50-mL portions of dilute nitric acid. Drain the aqueous phases into the beaker containing the first extract.

9.5 Sulfur compounds are not normally present in polybutenes. However, if present in amounts which may affect chlorine results significantly, the following procedure is recommended for removal of the sulfur compounds: Charge the aqueous phase from **9.4** to a separatory funnel and add 15 mL of ethyl ether to the mixture. Shake the contents of the funnel vigorously for 1 min, venting to the air frequently. Allow the contents of the funnel to stand until the two layers separate, then draw off the aqueous layer into a beaker. Extract the organic layer with two 15-mL portions of water, add the aqueous extracts to the beaker, and discard the organic layer. Add a few millilitres of 30 % hydrogen peroxide solution, heat the contents of the beaker on a steam hot plate until the ether has evaporated, then boil for 5 min, and cool to room temperature. Proceed in accordance with **9.6**.

9.6 Place the beaker on the titration stand, and insert the electrode system. Start the stirrer and record initial reading. Titrate slowly with 0.025 N AgNO_3 solution, recording readings after the addition of each drop of silver nitrate solution. Continue titrating until the point of maximum change in millivolt or pH scale reading is passed. Plot the volume of silver nitrate as abscissa and voltage or pH reading as ordinates. The end point is selected at the point of inflection of the curve.

NOTE 4—If the chloride content is known to be high, larger increments may be added until the titration is within 0.3 mL of the expected end point.

9.7 Blank—Titrate the same volume of solvent without the sample as a blank.

10. Calculation

10.1 Calculate the amount of total chlorine as follows:

$$\text{Total chlorine, ppm} = [(A - B)N/W] \times 35.460$$

where:

- A** = millilitres of AgNO_3 solution required for titration of the sample,
- B** = millilitres of AgNO_3 solution required for titration of the blank,

⁴ Organic Halogen Reagent (Sodium Biphenyl Solution) from Southwestern Analytical Chemicals, 209 Pleasant Valley Road, Austin, TX 78704, or the Aldrich Chemical Co., 1001 W. St. Paul Ave., Milwaukee, WI 53233 has been found satisfactory. Preparation of sodium biphenyl solution is described in McCoy, *The Inorganic Analysis of Petroleum*, Chemical Publishing Co., Inc., 212 Fifth Ave., New York, NY 10010, p. 127. (This book is no longer in publication.)

N = normality of the AgNO_3 solution, and
 W = grams of sample used.

11. Precision and Bias

11.1 *Precision*—This statement is provided for two approximate ranges of results. Insufficient samples were used in the inter-laboratory studies shown in Annex A1 to provide a precision statement encompassing the expected range of results.

11.1.1 Precision for test results near 14 ppm:

11.1.1.1 The repeatability standard deviation has been found to be 3 ppm. Therefore, the results of two properly conducted tests on the same sample by the same operator should not differ by more than 8 ppm.

11.1.1.2 The reproducibility standard deviation has been found to be 4 ppm. Therefore, the results of two different

laboratories on identical samples should not differ from each other by more than 12 ppm.

11.1.2 Precision for test results near 60 ppm:

11.1.2.1 The repeatability standard deviation has been found to be 3 ppm. Therefore, the results of two properly conducted tests on the same sample by the same operator should not differ by more than 9 ppm.

11.1.2.2 The reproducibility standard deviation has been found to be 9 ppm. Therefore, the results of two different laboratories on identical samples should not differ from each other by more than 25 ppm.

NOTE 5—The above precision statements were determined on polybutene samples with a viscosity of about 600 cSt at 100°C (210°F).

12. Keywords

12.1 chlorine; electrical; insulating; insulation; polybutenes

ANNEX

(Mandatory Information)

A1. PRECISION DATA

A1.1 Precision data for this test method are tabulated in **Table A1.1**

TABLE A1.1 Chlorine^A in Polybutene Round Robin No. 3

Sample	Series No.	Test No.	Laboratory 1	Laboratory 2	Laboratory 3	Laboratory 4	Laboratory 5	Laboratory 6
D	1	1	22	15	11	13	10	13
		2	15	18	12	14	14	21
	2	3	19	10	11	17	5	14
		4	18	13	11	16	8	18
E	1	1	59	38	63	58	65	61
		2	56	43	63	59	67	61
	2	3	65	45	64	59	67	67
		4	65	48	67	66	66	67

^AResults expressed in parts per million (ppm).

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