Designation: D 2522 – 95

Standard Test Method for Chlorine Content of Polybutenes Used for Electrical Insulation [1]

This standard is issued under the fixed designation D 2522; 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 This test method describes the determination of the total chloride content of polybutenes used for electrical insulation.

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.* For specific hazards information, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 878 Test Method for Inorganic Chlorides and Sulfates in Insulating Oils [2]

D 1193 Specification for Reagent Water [3]

D 2296 Specification for Continuity of Quality of Electrical Insulating Polybutene Oil for Capacitors [2]

3. Summary of Test Method

3.1 Organically bound chlorine is converted into sodium chloride by reaction with sodium biphenyl solution. The 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 D 878.

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 *Potentiometeric 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. [4] 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.

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 D 1193.

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.

---

[1] This test method is under the jurisdiction of ASTM Committee D-27 on Electrical Insulating Liquids and Gasesand is the direct responsibility of Subcommittee D27.06on Chemical Test.

Current edition approved March 15, 1995. Published May 1995. Originally published as D 2522 – 69. Last previous edition D 2522 – 89.

[2] *Annual Book of ASTM Standards*, Vol 10.03.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *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.

7.7 *Sodium Biphenyl Solution*—Transfer 300 mL of dry toluene and 58 g of metallic sodium to a 20 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.) [5]

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. [5] (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,
N = normality of the $AgNO_3$ solution, and
W = grams of sample used.

11. Precision and Bias

11.1 *Precision*:

11.1.1 *Repeatability*— Two duplicate determinations, made in the same laboratory on the same day, should not be considered suspect unless they differ by more than 8 ppm.

11.1.2 *Reproducibility*— The average of two duplicate determinations made in the same day, on the same sample by two different laboratories should not be considered suspect unless they differ by 9 ppm.

NOTE 5—The above precision statements were determined on polybutene samples with a viscosity of about 600 cSt at 100°C (210°F) and ranging in chlorine content from 15 to 65 ppm.

11.2 The bias of this test method cannot be determined because there is no accepted reference material.

12. Keywords

12.1 chlorine; electrical; insulating; insulation; polybutenes

[5] 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.)

APPENDIX

(Nonmandatory Information)

X1. PRECISION DATA

X1.1 Precision data for this test method are tabulated in Table X1.1.

TABLE X1.1 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

[A] Results expressed in parts per million (ppm).