Designation: D4301 – 05 (Reapproved 2013)

Standard Test Method for Total Chlorine in Epoxy Resins and Compounds[1]

This standard is issued under the fixed designation D4301; 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 a procedure for the determination of total chlorine in epoxy resins and glycidyl ethers.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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 hazard statements, see Section 8.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D6440 Terminology Relating to Hydrocarbon Resins

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *Other Document:*

OSHA Regulations, 29 CFR paragraphs 1910.1000 and 1910.1200 [3]

3. Terminology

3.1 *Definitions:* For definitions of terms used in this standard, See Terminology D6440.

4. Summary of Test Method

4.1 The material, dissolved in dimethoxyethane (DME) or other suitable inert solvent, is reacted with sodium biphenyl to convert bound organic chlorine to the water soluble chloride. The excess reagent is decomposed with isopropyl alcohol. The chloride ion is then titrated potentiometrically with silver nitrate.

5. Significance and Use

5.1 The presence of residual chlorine in epoxy resins is deleterious to final product properties. This test method has been found to be applicable to resins or ethers with chlorine contents ranging from 50 ppm to 35 % by weight. Other halogen compounds react with the reagent but are distinguished from chlorine by the final potentiometric titration. Epoxy and other functional groups will consume reagent but do not affect the results.

6. Apparatus

6.1 *Potentiograph.*

6.2 *Beaker,* of appropriate size.

6.3 *Buret,* of appropriate size.

6.4 *Stirrer,* magnetic or paddle.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used 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 water conforming to Type II of Specification D1193.

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

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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.

[3] A suitable mechanical holder is available from the Gardner Laboratory, Inc., 5521 Landy Lane, Washington, DC, Item 660.

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

7.3 *Sodium Biphenyl Reagent* [5] (in ether solution).

NOTE 1—Store in a refrigerator when not in use to prevent loss of activity.

7.4 *Nitric Acid* (HNO_3) (1 + 1), aqueous solution.

7.5 *1,2-Dimethoxyethane.*

7.6 *Silver Nitrate* ($AgNo_3$), 0.1 and 0.01 *N* standardized solutions.

7.7 *Methyl Red Indicator* (0.2 % alcohol solution), dissolve 0.2 g of methyl red in 100 mL of methanol, ethanol, or isopropanol.

7.8 *Isopropyl Alcohol.*

8. Hazards

8.1 Consult current OSHA Regulations, Supplier's Material Safety Data Sheets, and local regulations for all materials used in this test method.

9. Procedure

9.1 Pipet 5 mL of 1,2-dimethoxyethane into a clean, dry 250-mL beaker. Add a weighed amount of sample. (If the material is anticipated to be low in total chlorine, use 0.40 g of sample. For materials high in chlorine, weigh 0.1 to 0.2 g of sample.) Gently swirl to dissolve.

9.2 Add about 15 mL (one bottle) of sodium biphenyl reagent and mix thoroughly. If the solution loses its dark blue or green color within 5 min, add another 15 mL of reagent. Allow to react for 5 min.

9.3 Add 100 mL of isopropanol to decompose the excess reagent and to serve as the titrating medium. Add 3 to 5 drops of methyl red solution and neutralize with HNO_3 (1 + 1). Add 5 to 8 drops of acid in excess; start the stirrer.

9.4 Titrate the solution potentiometrically using standard $AgNO_3$ solution. (For low chlorine content, titrate with 0.01 *N* $AgNO_3$ and for high chlorine content use 0.10 *N* $AgNO_3$). The scale of the titration curve will depend on the instrument used but should give a curve of the approximate scale of 0.25 mL/cm and 30 mV/cm. The end point is selected as the middle of the steepest portion of the curve.

NOTE 2—It may be preferable to use the first or second derivative to determine the end point providing appropriate equipment is available.

9.5 Repeat the determination and also run a blank using all reagents but omitting the specimen.

10. Calculation

10.1 Calculate the weight percent of chlorine, *C*, as follows:

$$C = \frac{(V - B) \times N \times 3.546}{S}$$

where:

V = titration of specimen, mL,
B = titration of blank, mL,
N = normality of $AgNO_3$ solution, and
S = weight of specimen, g.

10.2 Calculate the mean of the two runs.

11. Precision

11.1 On the basis of an interlaboratory study, in accordance with Specification E691, of this test method in which one operator in each of three laboratories analyzed in duplicate on two different days five materials containing 0.1 to 0.3 % total chlorine, for a total of 60 determinations, the within-laboratory standard deviation was found to be 5 % relative and the between-laboratories standard deviation 7 % relative. Based on these standard deviations the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.2 *Repeatability*—Two results, each the mean of two runs, obtained by the same operator should be considered suspect if they differ by more than 13.9 % relative.

11.3 *Reproducibility*—Two results, each the mean of two runs, obtained by operators in different laboratories should be considered suspect if they differ by more than 19.4 % relative.

12. Keywords

12.1 chlorine; glycidyl ethers; liquid epoxy resins

[5] The sole source of supply of the reagent, (in small bottles (15-mL)) known to the committee at this time is Southwestern Analytical Chemicals, P. O. Box 485, Austin 63, TX. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend. It may also be prepared as described in *Analytical Chemistry*, Vol 26, p. 748.