



Designation: D4301 – 05

Standard Test Method for Total Chlorine in Epoxy Resins and Compounds¹

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 *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 1910.1200³

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.

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.

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**.

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

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.

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.

*A Summary of Changes section appears at the end of this standard