



Standard Test Methods for Epoxy Content of Epoxy Resins¹

This standard is issued under the fixed designation D 1652; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover the quantitative determination of the epoxy content of epoxy resins.

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 6.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 200 Practice for Preparation, Standardization, and Storage of Standard Solutions for Chemical Analysis³

3. Summary of Test Method

3.1 The resin is dissolved in a suitable solvent and the resulting solution is titrated with hydrogen bromide either directly or in situ. The hydrogen bromide reacts stoichiometrically with epoxy groups to form bromohydrins; therefore, the quantity of acid consumed is a measure of the epoxy content.

3.1.1 In Test Method A, the titration is direct with a standard solution of hydrogen bromide in glacial acetic acid.

3.1.2 In Test Method B, the titration is with standard perchloric acid in the presence of an excess of tetraethylammonium bromide. Hydrogen bromide generated in situ by the addition of perchloric acid to the quaternary ammonium halide rapidly opens the oxirane ring.

4. Significance and Use

4.1 The epoxy content of epoxy resins is an important variable in determining their reactivity and the properties of coatings made from them. These test methods may be used to determine the epoxy content of manufactured epoxy resins and

confirm the stated epoxy content of purchased epoxy resins.

5. Reagents

5.1 *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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

6. Hazards

6.1 Hydrogen bromide and glacial acetic acid are corrosive. Chlorobenzene and chloroform are considered hazardous. In addition to other precautions, take care to avoid inhalation and skin or eye contact with these chemicals. Use goggles or a face shield, or both. Protect skin by use of suitable protective clothing. All specimen preparations shall be done in a well ventilated area, such as a fume hood.

TEST METHOD A

7. Apparatus

7.1 *Buret*, closed-reservoir type. The buret tip should be fitted with a rubber stopper of proper size to fit the neck of the Erlenmeyer flask and the stopper should have an additional small hole to permit escape of replaced air during titration.

7.2 *Magnetic Stirrer*, adjustable speed.

7.3 *Magnetic Stirring Bars*, polytetrafluoroethylene (PTFE) coated.

8. Reagents and Materials

8.1 *Chlorobenzene* (**Warning**—See Section 6).

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

Current edition approved June 10, 1997. Published October 1997. Originally published as D 1652 – 59 T. Last previous edition D 1652 – 90 (1996).

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

³ *Annual Book of ASTM Standards*, Vol 15.05.

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.