



Designation: D 1726 – 90 (Reapproved 1996)

Standard Test Methods for Hydrolyzable Chloride Content of Liquid Epoxy Resins¹

This standard is issued under the fixed designation D 1726; 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 These test methods cover the determination of the easily hydrolyzable chloride content of liquid epoxy resins which are defined as the reaction product of a chlorohydrin and a di- or polyfunctional phenolic compound.

1.1.1 In Test Method A, the easily hydrolyzable chloride is saponified with potassium hydroxide and directly titrated with hydrochloric acid. This test method can be used for concentrations of 1 weight % and below.

1.1.2 In Test Method B, the easily hydrolyzable chloride is again saponified with potassium hydroxide, then titrated potentiometrically with silver nitrate. This test method can be used for concentrations of 5 to 2500 ppm hydrolyzable chloride.

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 Sections 8 and 14.

2. Referenced Documents

2.1 *ASTM Standards:*

D 841 Specification for Nitration Grade Toluene²

D 1193 Specification for Reagent Water³

3. Summary of Test Method

3.1 The sample is refluxed in the presence of a known amount of 0.1 N alcoholic potassium hydroxide. The amount of potassium hydroxide consumed in the hydrolysis is a measure of the hydrolyzable chloride content of the resin.

4. Significance and Use

4.1 The hydrolyzable chloride content of liquid 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 hydrolyzable chloride content of

manufactured epoxy resins and confirm the stated hydrolyzable chloride content of purchased epoxy resins.

5. Apparatus

5.1 *Reflux Apparatus*, consisting of a 250-mL Erlenmeyer flask attached to a reflux condenser and a hot plate with variable heat control.

6. Purity of Reagents

6.1 *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,⁴ where such specifications are available. 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.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

TEST METHOD A

7. Reagents and Materials

7.1 *Hydrochloric Acid, Standard (0.1 N)*—Dilute 9 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) to 1 L with water. Standardize against 0.25 g of sodium carbonate (Na₂CO₃) accurately weighed and dissolved in 75 to 100 mL of water.

7.2 *Methyl Ethyl Ketone*.

7.3 *Phenolphthalein Indicator Solution*—Dissolve 1 g of phenolphthalein in 100 mL of methanol, ethanol, or isopropanol.

7.4 *Potassium Hydroxide, Alcohol Solution (0.1 N)*—Dissolve 5.5 to 6.0 g of potassium hydroxide (KOH) in 1 L of methanol (99 %) or ethanol conforming to Formula No. SD-30 of the U.S. Bureau of Internal Revenue. No standardization of the solution is necessary.

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

Current edition approved May 25, 1990. Published July 1990. Originally published as D 1726 – 60. Last previous edition D 1726 – 87.

² *Annual Book of ASTM Standards*, Vol 06.04.

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

7.5 *Toluene* (**Warning**—See Section 8.), conforming to Specification D 841.

8. Hazards

8.1 Hydrochloric acid and potassium hydroxide are corrosive. Toluene and methyl ethyl ketone are flammable and their vapors can be harmful. Precautions should be taken to avoid inhalation and skin or eye contact with these chemicals. All sample preparations should be done in a well ventilated area, such as a fume hood.

9. Procedure

9.1 Weigh to the nearest 1 mg, 6 to 8 g of neutral specimen into a 250-mL glass-stoppered Erlenmeyer flask. By means of a pipet, transfer 50.0 mL of 0.1 *N* alcoholic KOH solution into the flask and add 15 mL of toluene. Stopper the flask and swirl to mix. Add a few boiling aids and attach to the reflux condenser on the hot plate.

9.2 Allow the solution to reflux gently for 15 ± 1 min. At the end of the reflux period, remove the flask from the hot plate and cool to room temperature with the condenser in place.

9.3 Remove the condenser, add 3 drops of phenolphthalein indicator solution to the specimen, and titrate with 0.1 *N* HCl. The end point is taken when 1 drop changes the solution from pink to colorless.

NOTE 1—Add 100 mL of methyl ketone to the specimen if required to ensure a homogeneous solution during titration of the excess KOH solution.

9.4 Make a blank determination on the reagents following the same procedure but omitting the specimen.

10. Calculation

10.1 Calculate the weight percent hydrolyzable chloride content, *H*, of the specimen as follows:

$$H = [(V - B)N \times 3.55]/W \quad (1)$$

where:

- B* = HCl required for titration of the blank, mL,
- V* = HCl required for titration of the hydrolyzed specimen, mL,
- N* = normality of the HCl,
- 3.55 = grams of chlorine per milliequivalent multiplied by the percentage factor of 100, and
- W* = specimen weight, g.

11. Precision

11.1 The following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 0.02 % absolute.

11.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 0.05 weight %.

TEST METHOD B

12. Apparatus

12.1 *Reflux Apparatus*, consisting of a 250-mL Erlenmeyer flask attached to a reflux condensor.

12.2 *Hot Plate*, with variable heat control.

12.3 *Magnetic Stirrer*, with polytetrafluoroethylene (PTFE)-coated stirring bar.

12.4 *Buret or Automatic Titrator*.

12.5 *Silver Electrode* or equivalent.

12.6 *Boiling Chips*.

13. Reagents and Material

13.1 *Acetone*.

13.2 *Bromcresol Green Indicator Solution (0.1 %)*—Dissolve 0.1 g of bromcresol green in 100 mL of water.

13.3 *Nitric acid*, (HNO₃) (1 + 1), diluted with water.

13.4 *Potassium Hydroxide*, alcohol solution (0.1 *N*)—Dissolve 5.6 g of potassium hydroxide (KOH) in 1 mL of methanol (99 %) or ethanol conforming to Formula No. SD-30 of the US Bureau of Internal Revenue. No standardization of the solution is necessary.

13.5 *Silver Nitrate*, alcohol solution (0.0025 *N*)—Dissolve 0.425 g of silver nitrate (AgNO₃), weighed to the nearest 1 mg, in 1 L of methanol (99 %) or ethanol conforming to Formula No. SD-30 of the US Bureau of Internal Revenue. Standardize against hydrochloric acid or sodium chloride solution (with traceability to an NIST standard).

13.6 *Toluene*.

14. Hazards

14.1 Nitric acid, potassium hydroxide, and silver nitrate are corrosive. Acetone and toluene are flammable and their vapors can be harmful. Precautions should be taken to avoid inhalation and skin or eye contact with these chemicals. All sample preparations should be done in a well ventilated area, such as a fume hood.

15. Procedure

15.1 Weigh 2 to 3 g of specimen, to the nearest 0.005 g, into a 250-mL Erlenmeyer flask. Add 20 mL of toluene, 20 mL of acetone, and 50 mL of 0.1 *N* alcoholic KOH. Swirl or mix until dissolution is complete.

15.2 Add several boiling chips, connect the flask to the reflux condenser, and gently reflux for 15 ± 1 min on a hot plate.

15.3 Remove the hot plate from under the flask and allow the flask and contents to cool to room temperature. Rinse down the condenser with acetone then remove from the flask.

15.4 Quantitatively transfer the contents of the flask to a 250-mL beaker using acetone as wash solution. Dilute the solution to about 125 mL with acetone, insert a stirring bar, and place on a magnetic stirrer.

15.5 Add five drops of bromcresol green indicator. While stirring add 1 + 1 nitric acid dropwise just until the permanent color changes from blue to yellow.

NOTE 2—**Caution**—Do not add excess nitric acid. Do not acidify the solution until ready to begin the titration. Make certain that the solution is at room temperature before acidifying. These cautions are necessary to prevent the chloride results from being low due to recombination with the resin.

15.6 Titrate with 0.0025 *N* silver nitrate using the combination silver electrode and the automatic titration buret.