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Standard Guide for Testing Epoxy Resins¹

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^{ε1} NOTE—Keywords were added editorially in October 1996.

1. Scope

1.1 This guide covers methods for testing epoxy resins as listed in Table 1. All of the methods were tested by interlaboratory participation in accordance with usual ASTM guidelines. Each method specifies a recommended amount of sample for starting a separate analysis, but several of the procedures can be conducted on the same starting material if so desired. For example, viscosity, color, and density could be run on the same specimen.

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.*

2. Referenced Documents

2.1 ASTM Standards:

- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)²
- D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)³
- D 1259 Test Methods for Nonvolatile Content of Resin Solutions⁴
- D 1475 Test Method for Density of Paint, Varnish, Lacquer, and Related Products⁴
- D 1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)⁴
- D 1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method⁵
- D 1639 Test Method for Acid Value of Organic Coating Materials⁵
- D 1652 Test Method for Epoxy Content of Epoxy Resins⁵
- D 1726 Test Method for Hydrolyzable Chlorine Content of Liquid Epoxy Resins⁵

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² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 06.04.

⁴ Annual Book of ASTM Standards, Vol 06.01.

⁵ Annual Book of ASTM Standards, Vol 06.03.

TABLE 1 Methods for Testing Epoxy Resins

Test Method	Section	ASTM Designation
Epoxy content	4	D 1652
Hydrolyzable chlorine	5	D 1726
Total chlorine	6	D 1847
Viscosity	7	D 445, D1545
Color	8	D 1544, D1209
Density	9	D 1475
Nonvolatile content	10	D 1259
Acid value	11	D 1639

D 1847 Test Methods for Total Chlorine Content of Epoxy Resins⁵

3. Significance and Use

3.1 This guide directs the user to test methods that determine properties generally accepted as standard test items for classification of epoxy resins.

4. Epoxy Content

4.1 The epoxy content of epoxy resins is determined by reacting a solution of the resin with a standard solution of hydrogen bromide in glacial acetic acid. The quantity of acid consumed is a measure of the epoxy content. Test Methods D 1652 was found to have a repeatability of 2 % of the epoxy content and a reproducibility of 6 % of the epoxy content.

5. Hydrolyzable Chlorine

5.1 Test Method D 1726 covers the determination of the easily hydrolyzable chlorine content of liquid epoxy resins in concentrations below 1 weight %. The specimen is refluxed with a known amount of a standard alcoholic potassium hydroxide solution. The amount of hydroxide consumed is measured by titration and corresponds to the hydrolyzable chlorine content of the resin. By interlaboratory testing, the repeatability was found to be 0.02 %, and the reproducibility was found to be 0.05 %, both absolute.

6. Total Chlorine

6.1 Organic and inorganic chlorine compounds are determined in epoxy resins in accordance with Test Methods D 1847. The resin specimen is oxidized by combustion in a bomb containing oxygen under pressure. The chlorides formed are dissolved in a sodium carbonate solution and then either titrated or determined gravimetrically. The absolute repeatability and reproducibility for the titration method are 0.02 and

0.05 weight %, respectively. For the gravimetric method, the repeatability and reproducibility are both reported to be 0.05 weight %, absolute.

7. Viscosity

7.1 Kinematic viscosity is determined by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer in accordance with Test Method D 445. The dynamic viscosity can be obtained by multiplying the measured kinematic viscosity by the density of the liquid. The limits of precision at the three sigma level of confidence were found to be 0.35 % relative within one laboratory and 0.70 % relative between laboratories.

7.2 It is also possible to measure viscosity by the bubble time method described in Test Method D 1545. The rise time of a bubble in the resin contained in an ASTM viscosity tube is measured. The rise time in seconds is approximately equal to the viscosity in stokes. In the range from 4.5 to 440 St, duplicate runs by one operator should be considered suspect if they differ by more than 4.9 % relative. Two results, each the mean of duplicates obtained by different operators should be considered suspect if they differ by more than 9.0 % relative.

8. Color

8.1 Test Method D 1544 covers the measurement of the color of transparent liquids by means of comparison with arbitrarily numbered glass standards. Two results obtained by a single operator should be considered suspect if they differ by more than two thirds of a color number. Two results, each the mean of duplicate measurements obtained by operators in different laboratories should be considered suspect if they differ by more than four thirds of a color number.

8.2 The color of epoxy resins having light absorption characteristics nearly identical with those of the platinum-cobalt color standards can be measured by the use of Test Method D 1209.

9. Density

9.1 Test Method D 1475 is applicable to liquid epoxy resins. This test method makes use of a weight-per-gallon cup and is particularly useful where the fluid to be measured has a high viscosity or where a component is too volatile for a specific gravity balance determination. The limits of precision at the three sigma level of confidence were found to be ± 0.008 lb/gal within one laboratory and ± 0.015 lb/gal between laboratories.

10. Nonvolatile Content

10.1 Since epoxy resins tend to release solvent slowly, it is preferred to use a thin film or “foil method,” in which the resin is pressed between sheets of aluminum foil to spread it to a thin film. Method B of Test Methods D 1259 is applicable to epoxy resins. The repeatability is 0.3 % and the reproducibility is 0.5 %, both absolute.

11. Acid Value

11.1 Acid value of an epoxy resin as determined by Test Method D 1639 is expressed as the number of milligrams of potassium hydroxide required to neutralize the free acidity of 1 g of nonvolatile material under the condition of the test.

12. Keywords

12.1 epoxy resins

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