
Test Procedure for**DETERMINING EPOXIDE EQUIVALENT AND AMINE
VALUE OF RESINS****TxDOT Designation: Tex-815-B****Effective Date: May 2000**

1. SCOPE

- 1.1 Use this method to determine the epoxide equivalent for epoxy resins and the amine value for polyamide resins.
 - 1.2 Reference ASTM D 1652 and D 2074 (modified by D-9-J).
 - 1.3 The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.
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2. APPARATUS

- 2.1 *Analytical balance*, Class B in accordance with Tex-901-K.
 - 2.2 *Buret*, 25 mL (0.85 fl. oz.), for titration.
 - 2.3 *Erlenmeyer flasks*, 50 mL (1.7 fl. oz.), with stoppers.
 - two for duplicate titration used in standardization of HBr solution
 - two for duplicate titration used for each sample of epoxy or polyamide resin.
 - 2.4 *Two beakers*, 100 mL (3.4 fl. oz.)
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3. MATERIALS

- 3.1 *Hydrobromic Acid (HBr)*, 47–49%, reagent grade.
 - 3.2 *Acetic acid*, glacial, reagent grade.
 - 3.3 *Potassium acid phthalate (Pht)*, reagent grade.
 - 3.4 *Methyl violet indicator*, 0.1 g in 100 mL (3.4 fl. oz.) glacial acetic acid.
 - 3.5 *Methyl isobutylketone (MIBK)*, reagent grade.
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Note 1—The presence of water at any time in this procedure will invalidate the results. Cloudiness in the sample or test solutions indicates the presence of water.

4. PROCEDURES

4.1 *Standardizing Titrant:*

4.1.1 Prepare the 0.1 N HBr in glacial acetic acid by adding 8 g of HBr to 500 mL (16.9 fl. oz.) of acetic acid.

4.1.2 Analytically weigh 0.4 ± 0.005 g of potassium acid phthalate into a 50-mL (1.7-fl. oz.) Erlenmeyer flask. Weigh a duplicate into a second flask.

4.1.3 Add 10 mL (0.33 fl. oz.) of glacial acetic acid to each sample and heat gently to dissolve the phthalate.

4.1.4 Titrate the sample, using approximately 12.5 mL (0.42 fl. oz.) of the 0.1 N HBr solution, to a green color using two to three drops of methyl violet indicator. (See Section 5.)

4.1.4.1 Use the same amount of indicator for each sample because of the small amount of solvent.

4.1.4.2 The solution will change from purple to dark, then light, blue until obtaining the green color. The final color change is obvious.

4.2 *Determining Epoxy Equivalent or Amine Value:*

4.2.1 Analytically weigh 1.0 ± 0.001 g of the 75% solids epoxy, 0.4 ± 0.001 g 100% solids epoxy, or 0.4 ± 0.001 g of the 70% solids polyamide sample into a 100-mL (3.4-fl. oz.) beaker (0.75 or 0.35 g for a 100% solids sample).

4.2.1.1 Use a quantity of sample that will yield 0.001–0.002 g equivalents of epoxy groups. (See Section 5.)

4.2.1.2 Also run a duplicate sample.

4.2.2 Dissolve the samples in 10 mL (0.35 fl. oz.) of MIBK. Just prior to titration, add 10 mL (0.35 fl. oz.) glacial acetic acid to the sample, and titrate with the standardized HBr to the same color as the standards. The endpoint for the amine value is not the same as for the epoxide procedure. The amine value solution turns milky, then green.

5. CALCULATIONS

5.1 Calculate the normality of the HBr solution:

$$\text{Normality of HBr Solution} = \frac{\text{Weight of Pht} \times 1000}{204.2 \times \text{mL of HBr}}$$

5.2 Calculate the epoxy equivalent of epoxy resins:

$$\text{Epoxy Equivalent} = 1000 \bullet \frac{\text{Weight of Sample} \times \% \text{ NVM (fraction)}}{\text{Normality of Hbr} \times \text{mL of Hbr}}$$

5.3 Calculate the amine value of polyamide resins:

$$\text{Amine Value} = \frac{\text{mL of HBr} \times \text{Normality of HBr} \times 56.1}{\text{Weight of Polyamide} \times \% \text{ NVM (fraction)}}$$

6. ARCHIVED VERSIONS

6.1 Archived versions are available.