Method of Test for

# DETERMINATION OF EPOXY EQUIVALENT WEIGHT OF EPOXY RESINS AND SOLUTIONS

DOTD Designation: TR 518-93

## I. Scope

This method of test determines the epoxy equivalent weight of epoxy resins and solutions. The epoxy equivalent weight is significant since the physical properties of resins such as flexibility, brittleness, permeability, and adhesion are directly related to this chemical parameter.

## II. Health Precautions

This procedure utilizes hazardous chemicals and solutions. Proper handling procedures and ventilation are mandatory.

## III. Apparatus

A. **Adjustable speed magnetic stirrer**
B. **Analytical balance** - accurate to 0.0001g
C. **Glass beakers** - 50 mL, 150 mL, 250 mL, and 500 mL in size
D. **Graduated cylinders** - 10 mL and 50 mL, in size
E. **Magnetic stirring bars**
F. **Microburette** - 10 mL
G. **Volumetric flask** - 1000 mL
H. **Hot plate**
I. **Oven** - capable of maintaining 100°C (212°F)
J. **Worksheet** (Figures 1 & 2)

## IV. Reagents and Solutions

A. Reagents
   1. Acetic anhydride $(CH_3CO)_20$
   2. Crystal violet indicator $(C_{25}H_{30}ClN_3)$
   3. Glacial acetic acid $(CH_3COOH)$
   4. Methylene chloride $(CH_2Cl_2)$
   5. Perchloric acid $(HClO_4)$ 72%
   6. Potassium hydrogen phthalate $(HOOCC_6H_4COOK)$
   7. Tetraethylammonium bromide $(NEt_4Br)$

B. Solutions
   1. Crystal violet indicator solution - Prepare a 0.1% solution of crystal violet in glacial acetic acid by dissolving 0.1 g of crystal violet in 100 mL of glacial acetic acid in a 250 mL beaker.
   2. Perchloric acid solution - Prepare a 0.1N solution of perchloric acid in glacial acetic acid by adding 8.5 mL of 72% perchloric acid and 20 mL of acetic anhydride to 400 mL of glacial acetic acid in a 1000 mL volumetric flask. Shake well and dilute to full mark with glacial acetic acid.
   3. Tetraethylammonium bromide solution - Prepare the indicator solution by dissolving 100 g of tetraethylammonium bromide in 400 mL of glacial acetic acid. Add 3-5 drops of crystal violet indicator solution.

## V. Test Samples & Specimens

A. Samples - shall consist of the vehicle portion of paints or small quantities of epoxy resin systems.
B. Specimens - shall be approximately 1g.

## VI. Solution Standardization and Procedure

A. Standardization of Perchloric Acid Solution
   1. Weigh approximately 0.2 g of potassium hydrogen phthalate which has been previously dried overnight at 100°C (212°F) into a 150 mL beaker on an analytical balance.

2. Record the weight to 0.0001 g as K (Figure 1 or Figure 2).
3. Add 50 mL of glacial acetic acid and warm on low heat until the sample is dissolved.
4. Allow to cool and use an additional 50 mL of glacial acetic acid to wash down the side of the beaker.
5. Add 3-5 drops of crystal violet indicator.
6. Insert a stirring bar into the beaker and stir while titrating with the perchloric acid to a yellow green end point and record the amount of titrant added as P to the nearest .05 mL.

B. Procedure for Determining Epoxy Equivalent Weight

1. Weigh approximately 1 g of the test specimen in a 50 mL beaker.
2. Record the specimen weight to 0.0001 g as A.
3. Add 10 mL of methylene chloride and 10 mL of tetraethylammonium bromide indicator solution.
4. Add 5-6 drops of crystal violet indicator.
5. Insert a stirring bar into the beaker and stir while titrating with the standardized perchloric acid until the color changes from crystal violet to green.
6. Record the volume to the nearest 0.1 mL of perchloric acid titrant used to reach the end point as B.

## VII. Calculations

A. Calculate the normality of the perchloric acid solution (N) using the following formula:

$$N = \frac{K \times 1000}{204.2 \times P}$$

where:

K = weight of potassium hydrogen phthalate, g
P = volume of perchloric acid solution, mL
1000 = constant, mL to L
204.2 = constant, chemical factor

example:

K = 0.2156 g
P = 6.25 mL

$$\frac{0.2156 \times 1000}{204.2 \times 6.25} = \frac{215.60}{1276.25}$$

$$= 0.16893$$

$$N = 0.169$$

B. Calculate the epoxy equivalent weight ($E_w$) for resins and solutions using the following formula:

$$E_w = \left(\frac{A \times 1000}{B \times N}\right) \times \frac{D}{100}$$

*(Figure 1)*

or

$$E_w = \frac{A \times 1000}{B \times N}$$

*(Figure 2)*

where:

A = specimen wt, g
B = perchloric acid titer volume, mL
N = normality of perchloric acid (from above)
D = % resin solids (determined by other test procedures if applicable)
1000 = constant, mL to L
100 = constant, for percentage

example:

A = 1.0017 g
B = 2.50 mL
N = 0.169
D = 22.5%

$$\left(\frac{1.0017 \times 1000}{2.50 \times 0.169}\right) \times \frac{22.5}{100}$$

$$= \frac{1001.7000}{0.4225} \times .2250$$

$$= 2370.8875 \times .2250$$

$$= 533.4496$$

$$E_w = 533$$

***NOTE 1:*** *In order to obtain the desired accuracy, the calculations should be carried to four decimal places as shown.*

## VIII. Report

Report the epoxy equivalent weight to the nearest whole number.

***NOTE 2:*** *Due to the variations in titrations, color indications and solids determinations, the repeatability of this test method should be considered suspect only for deviations of more than ten epoxy equivalent weights.*

## IX. Normal Test Reporting Time

Normal test reporting time is 3 days.

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## EPOXY EQUIVALENT CALCULATION (TR 518)

Normality of Perchloric Acid, $N = \frac{K \times 1000}{204.2 \times P}$ = 0.169

| | |
|---|---|
| Potassium hydrogen phthalate wt. + tare wt., g | 60.8581 |
| Tare wt., g | 60.6425 |
| Potassium hydrogen phthalate wt., g (K) | .2156 |
| Perchloric acid volume, mL (P) | 6.25 |

Epoxy Equivalent, $E_w = \left(\frac{A \times 1000}{B \times N}\right) \times \frac{D}{100}$ = 533

| | |
|---|---|
| Sample wt. + tare wt., g | 10.9028 |
| Tare wt., g | 9.9011 |
| Sample wt., g (A) | 1.0017 |
| Perchloric acid titer volume, mL (B) | 2.50 |
| Normality of perchloric acid, N (N) | 0.169 |
| Non-Volatile vehicle, % (D) *from "BASE" section of Organic Zinc Primer worksheet, Matcode 209* | 22.5 |

## AMINE VALUE CALCULATION (TR 519)

Normality of Perchloric Acid, $N = \left(\frac{K \times 1000}{204.2 \times P}\right)$ = _____

| | |
|---|---|
| Potassium hydrogen phthalate wt. + tare wt., g | |
| Tare wt., g | |
| Potassium hydrogen phthalate wt., g (K) | |
| Perchloric acid volume, mL (P) | |

Amine Value, $V = \left(\frac{A \times 56.1 \times N}{C \times D}\right) \times 100$ = _____

| | |
|---|---|
| Perchloric acid titer volume, mL (A) | |
| Normality of perchloric acid, N (N) | |
| Sample wt. + tare wt., g | |
| Tare wt., g | |
| Sample wt., g (C) | |
| Solids, % (D) *from "CURE" section of Organic Zinc Primer worksheet, Matcode 209* | |

Tested by: J. V. Date: 1/8/93

Checked by: J. D. B. Date: 1/8/93

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Figure 1

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EPOXY

EPOXY EQUIVALENT CALCULATION (TR 518)

Normality of Perchloric Acid, $N = \frac{K \times 1000}{204.2 \times P}$ = 0.143

| | |
|---|---|
| Potassium hydrogen phthalate wt. + tare wt., g | 60.8581 |
| Tare wt., g | 60.6425 |
| Potassium hydrogen phthalate wt., g (K) | .2156 |
| Perchloric acid volume, mL (P) | 7.40 |

Epoxy Equivalent, $E_w = \frac{A \times 1000}{B \times N}$ = 190

| | |
|---|---|
| Sample wt. + tare wt., g | |
| Tare wt., g | |
| Sample wt., g (A) | 0.2150 |
| Perchloric acid titer volume, mL (B) | 7.90 |
| Normality of perchloric acid, (N) | 0.143 |

FILLER CONTENT (ASTM C 881)

| | |
|---|---|
| Sample wt. + tare wt., g | |
| Tare wt., g | |
| Sample wt., g (A) | 1.0515 |
| Wt. of crucible after filtration, g | 22.7250 |
| Wt. of crucible before filtration, g | 22.5195 |
| Wt. of filler, g (B) | .2055 |

% Filler Content = $\frac{B}{A} \times 100$ = 19.5

Tested by: JV Date: 1/8/93

Checked by: Q.D.B. Date: 1/8/93

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Figure 2