Kentucky Method 64-264-08
Dated 03/31/08

TEST METHOD FOR ANALYSIS OF EPOXY CONTENT

1. SCOPE

1.1 This method is designed to quantitatively determine the epoxy content of the epoxy component of two component structural steel coatings.

2. MATERIALS AND REAGENTS

2.1 Balance accurate to 0.001g

2.2 Burette, 10 ml capacity with 0.05 ml graduations

2.3 Graduated cylinder, 25 ml

2.4 Erlenmeyer flasks, 125 ml

2.5 Volumetric flasks, 50 ml

2.6 Magnetic stirrer, adjustable speed

2.7 Magnetic stirring bars, PTFE coated

2.8 High speed centrifuge

2.9 Centrifuge tubes, 50 ml, disposable polypropylene

2.10 Xylene, reagent grade

2.11 Chloroform-Chlorobenzene mixture (1:1), reagent grade

2.12 Crystal Violet indicator solution

2.13 Glacial Acetic Acid, reagent grade

2.14 Hydrogen Bromide (HBr), 30% by weight in acetic acid (approx. 5.78 N)

2.15 Potassium acid phthalate ($KHC_8H_4O_4$), primary standard grade

2.16 Potassium acid phthalate ($KHC_8H_4O_4$) 0.1N solution

3. PREPARATION OF STANDARD SOLUTIONS

3.1 Crystal Violet Indicator Solution:
Dissolve 0.1 g crystal violet in 100 ml glacial acetic acid.

3.2 Potassium acid phthalate ($KHC_8H_4O_4$) 0.1N solution:
Dry the potassium acid phthalate in an oven @ 110°C to a constant weight; cool in dessicator. Dissolve 1 g (weighed to 0.001 g) potassium acid phthalate in 25 ml of glacial acetic acid by gently heating. Record weight. Cool to room temperature.

4. STANDARDIZATION OF HYDROGEN BROMIDE SOLUTION

4.1 The 30% HBr solution should be standardized each day.

4.2 To the potassium phthalate solution (see 3.2) add 10 drops of crystal violet indicator solution.

4.3 Titrate the potasium acid phthalate solution with the 30 % HBr solution to a blue-green end point.

5. SAMPLE PREPARATION: (Perform in duplicate)

5.1 To a tared centrifuge tube add 5 g sample, and record weight to 0.001 g.

5.2 Thin the sample with 5 ml xylene.

5.3 Tap the bottom of the centrifuge tube on the side of the counter to disperse the sample.

5.4 Centrifuge the sample to produce a relative centrifugal force (RCF) of 4200 for 30 minutes. See 8.1 for calculation of RCF.

5.5 Transfer the supernatant to the 125 ml Erlenmeyer flask in which the titration will be performed.

5.6 Re-disperse the pigment in the bottom of the centrifuge tube with 5 ml of xylene.

5.7 Centrifuge the sample again for 30 minutes as in 5.4.

5.8 Add this supernatant to the original supernatant for analysis.

6. PROCEDURE: (Perform in duplicate)

6.1 To the supernatant in the 125 ml Erlenmeyer flask add 25 ml of a 1:1

mixture of chloroform and chlorobenzene.

6.2 Place a magnetic stirring bar into the flask and mix on the magnetic stirrer to dissolve; stir at a moderate speed to avoid splashing.

6.3 Add 10 drops of crystal violet indicator solution.

6.4 Titrate with the 30 % HBr in acetic acid solution to a blue-green endpoint with the stirrer rotating at a moderate speed to avoid splashing.

6.5 Slow down the titration near the endpoint to allow ample time for the reaction to take place (see 7.1).

6.6 Record the amount of titrant used.

6.7 Make a blank determination on the reagent in an identical manner.

6.8 Record the amount of titrant used for the blank determination.

7. NOTES

7.1 After each addition allow time for the reaction to take place. The endpoint is a blue-green color. When approaching the endpoint additions should be dropwise. If the solution turns yellow the sample has been over-titrated.

8. CALCULATIONS

8.1 Calculation for Relative Centrifugal Force (RCF):

$$RCF = 0.00001118 \times r \times N^2$$

Where: RCF = Relative Centrifugal Force
r = Radius of Rotation
N = Revolutions Per Minute (RPM)

8.2 Calculation for Normality of the 30% HBr in acetic acid:

$$N = (W \times 1000)/(204.2 \times V)$$

Where: W = potassium acid phthalate used, g
V = HBr solution used, ml

8.3 Calculation for weight per epoxy equivalent (grams of that component which contains one gram equivalent of epoxy groups):

$$WPE = 1000W / N(V-B)$$

Where: V = HBr solution used for titration of the sample, ml
B = HBr solution used to titrate the blank, ml
N = normality of the HBr solution
W = weight of the sample used, g

9. REPORT

9.1 Report the weights of the duplicate coating samples to the nearest 0.001g.

9.2 Report the Normality of the HBr solution.

9.3 Report the WPE in duplicate in grams.

9.4 Results should be considered suspect if duplicate analyses differ by more than 5% relative percent difference when performed by a single analyst.

APPROVED _____
DIRECTOR
DIVISION OF MATERIALS

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