## TEST METHOD FOR THE ANALYSIS OF BASE NUMBER

## 1. SCOPE

1.1 This method is designed for the determination of the titratable alkalinity (Base Number) of the resin system found in calcium sulfonate structural steel coatings.

## 2. MATERIALS AND REAGENTS

- 2.1 Balance accurate to 0.001g
- 2.2 Hot plate
- 2.3 Burette, 50 ml capacity with 0.1 ml graduations
- 2.4 Erlenmeyer flasks, 250 ml wide-mouth
- 2.5 Beakers, 250 ml
- 2.6 Volumetric flask, 100 ml
- 2.7 Class A 50 ml pipet
- 2.8 Buchner filter funnel, 83mm
- 2.9 Whatman 42 filter papers, 70 mm
- 2.10 Magnetic stir bar
- 2.11 Glass boiling beads
- 2.12 Acetic Acid, 0.2 N
- 2.13 Ammonium Hydroxide, 0.2 N
- 2.14 Heptane, reagent grade
- 2.15 Isopropyl Alcohol, reagent grade
- 2.16 Ethanol, reagent grade

- 2.17 Distilled water, ASTM D1193 Type II
- 2.18 Neutral red-Bromothymol blue mixed indicator

#### 3. PREPARATION OF STANDARD SOLUTIONS

3.1 Neutral red-Bromothymol blue mixed indicator solution 0.05 g Neutral red and 0.05 g Bromothymol blue in 100 ml volumetric flask. Dilute to mark with ethanol and mix well.

## 4. SAMPLE PREPARATION

- 4.1 To a tared 250 ml Erlenmeyer flask add 1.0 g of sample. Record weight.
- 4.2 Effect solubility of the sample by the addition of 4 ml Heptane. Add glass boiling beads and swirl flask until sample is completely dissolved.
- 4.3 Pipet 50 ml 0.2N Acetic acid into flask.
- 4.4 Add 10 ml Isopropyl alcohol and 40 ml distilled water to the flask.
- 4.5 Place the flask on a hot plate and carefully bring to a boil for 5-10 minutes to distill the Heptane away.
- 4.6 Remove flask from hot plate and place in cool water bath; cool to room temperature.

#### 5. TITRATION PROCEDURE

- 5.1 Add 10 drops Neutral red-Bromothymol blue mixed indicator to the flask.
- 5.2 Titrate to a gray (colorless) endpoint using 0.2N Ammonium Hydroxide (See Note 6.1).

# 6. NOTES

- At the beginning, the solution will be rose colored. As the endpoint is approached the solution will turn colorless; this is the endpoint. If the sample is over-titrated the solution will turn a faint blue color.
- 6.2 For products with an expected Base Number of less than 150, increase sample size to 1.5-2.0 grams, and/or utilize 25 ml 0.2N Acetic acid.

## 7. CALCULATIONS

7.1 Calculation for Base Number
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$$\underbrace{ [(ml\ CH_3COOH\ x\ N\ CH_3COOH) - (ml\ NH_4OH\ x\ N\ NH_4OH)]}_{Sample\ wt} \quad x\ 56.1 = \ Base\ Number$$

## 8. REPORT

- 8.1 Report the weights of the duplicate coating samples to the nearest 0.001g.
- 8.2 Report the duplicate Base Number results.
- 8.3 Results should be considered suspect if duplicate analyses differ by more than 5% RPD when performed by a single analyst.

APPROVED

DIRECTOR
DIVISION OF MATERIALS

DATE

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