

TEST METHOD FOR ANALYSIS FOR AMINE CONTENT

1. SCOPE

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

2. MATERIALS AND REAGENTS

- 2.1 Mettler-Toledo DL50 titrator or equivalent
- 2.2 Balance accurate to 0.001g
- 2.3 High speed centrifuge
- 2.4 Centrifuge tubes, 50 ml, disposable polypropylene
- 2.5 Hot plate, with variable heat control
- 2.6 Magnetic stirrer, adjustable speed
- 2.7 Magnetic stirring bars, PTFE coated
- 2.8 250 ml low-form beakers
- 2.9 250 ml titration vessel
- 2.10 Graduated cylinders, 10 ml, 100 ml
- 2.11 Methylene chloride, reagent grade
- 2.12 Isopropyl alcohol (99%), reagent grade
- 2.13 Chloroform, reagent grade
- 2.14 Hydrochloric acid, conc., reagent grade
- 2.15 Hydrochloric acid, standard solution, 1.0 N
- 2.16 Distilled water, ASTM D1193 Type II

- 2.17 Sodium carbonate, reagent grade
3. PREPARATION OF STANDARD SOLUTIONS
- 3.1 Hydrochloric acid solution, 1.0 N:
Add 170 ml conc. Hydrochloric acid to 1000 ml distilled water in a 2L volumetric flask. Dilute to volume with distilled water and mix.
4. STANDARDIZATION OF 0.1 N HYDROCHLORIC ACID SOLUTION
- 4.1 Dry sodium carbonate to a constant weight in an oven at 110°C.
- 4.2 Weigh 0.53 g sodium carbonate to the nearest 0.001 g.
- 4.3 Dissolve sodium carbonate in 50 ml distilled water.
- 4.4 Using the titrator, titrate with the 1.0 N HCl to a pH of 3.5.
- 4.5 Record the ml of HCl used.
- 4.6 Calculate the normality of the HCl (see 7.2).
5. SAMPLE PREPARATION: Perform in duplicate
- 5.1 If the component to be tested is clear, it can be used in the method as is.
- 5.2 If the component to be tested is pigmented, the pigment must be removed..
- 5.2.1 Weigh 1.0 g of sample (to 0.001g) into a disposable polypropylene centrifuge tube.
- 5.2.2 Thin the sample with 3-4 ml methylene chloride; tap the bottom of the tube on the edge of the counter to disperse the sample.
- 5.2.3 Centrifuge the sample to produce a minimum relative centrifugal force (RCF) of 4200 for 30 minutes. See 7.1 for calculation of RCF.
- 5.2.4 Transfer the supernatant to a 250 ml low-form beaker.
- 5.2.5 Re-disperse the pigment in the bottom of the centrifuge tube with 3-4 ml methylene chloride.
- 5.2.6 Centrifuge the sample again for 30 minutes as in 5.2.3.
- 5.2.7 Add this supernatant to the original supernatant for analysis.

6. PROCEDURE: (Perform in duplicate)
- 6.1 Weigh 1.0 g to nearest 0.001 g into a 250 ml low-form beaker or if pretreatment was necessary, use all the supernatant from that procedure.
 - 6.2 Record the weight.
 - 6.3 Add 90 ml chloroform and 10 ml isopropyl alcohol to the beaker.
 - 6.4 Boil on a hot plate for 1 minute.
 - 6.5 Cool to room temperature.
 - 6.6 Pour contents into the 250 ml titration vessel.
 - 6.7 Attach correct pH electrode (Mettler-Toledo DG111-SC).
 - 6.8 Calibrate the pH electrode with standard pH 4.00 and pH 7.00 buffer solutions.
 - 6.9 Attach burette and bottle assembly containing the standardized 1.0 N HCl solution to the titrator.
 - 6.10 Bleed any air out of the burette and lines by putting titrator into "Dispense" mode and dispensing 5 ml while tapping on the burette and lines gently with a teflon stir stick. Repeat until there are no air bubbles.
 - 6.11 Attach titration vessel containing the prepared sample to the titrator; add enough isopropyl alcohol so that the end of the electrode remains immersed in the solution.
 - 6.12 Using user method "Amine" titrate the sample through the endpoint.
 - 6.13 Record the volume of standardized 1.0 N HCl used to the nearest 0.001 ml.
 - 6.14 Calculate the amine value (7.3).
 - 6.15 After each titration the electrode, feed line and stirrer should be cleaned with isopropyl alcohol.
 - 6.16 Soak the electrode in distilled water for 5 minutes before the next titration.
 - 6.17 Titrate the duplicate sample.

6.18 Calculate the relative percent difference (RPD)(see 7.4).

7. CALCULATIONS

7.1 Calculation for Relative Centrifugal Force (RCF):

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

Where: r = radius of rotation

N = revolutions per minute (RPM)

7.2 Calculation for Normality of the HCl solution:

$$N = (W \times 1000) / (53 \times V)$$

Where: N = normality of the HCl solution

W = sodium carbonate used, g

V = HCl used, ml

53 = sodium carbonate equivalent weight

7.3 Calculation for the amine value:

$$\text{amine value} = (V \times N \times 56.1) / S$$

Where: V = HCl required to titrate the sample, ml

N = normality of the HCl solution

S = sample weight used, g

8. REPORT

- 8.1 Report the weights of the duplicate coating samples to the nearest 0.001 g.
- 8.2 Report the normality of the HCl solution.
- 8.3 Report the amine value in duplicate.
- 8.4 Results should be considered suspect if duplicate analyses differ by more than 2% relative percent difference when performed by a single analyst.

APPROVED

DIRECTOR
DIVISION OF MATERIALS

DATE

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