



Transport
Roads & Maritime
Services

Test method T1025

Determination of the total amine value by
reference potentiometric method

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Revision Summary

Ed/Rev Number	Clause Number	Description of Revision	Authorisation	Date
		Reformatted and Revision Summary Added	D. Dash	Jun2001
Ed 2/ Rev 0	All	Reformatted RMS template	J Friedrich	November 2012

Note that Roads and Maritime Services is hereafter referred to as 'RMS'.

The most recent revision to Test method T1025 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T1025

Determination of the total amine value by referee potentiometric method

1. Scope

This test method sets out the procedure for determination of the total amine value of aggregate precoat and binder additives. This method conforms with that given in ASTM D2073-66.

2. Apparatus

- (a) A balance, of 200 g capacity, accurate and readable to 0.0001 g.
- (b) a pH meter with electrodes.
- (c) Magnetic stirrer and plastic coated stirrer bar.
- (d) Hot plate with variable control.
- (e) Microburette of capacity 10 mL and graduated to 0.02 mL.
- (f) A 1L volumetric flask.
- (g) A 100 mL and 500 mL graduated measuring cylinder.
- (h) A 50 mL burette.
- (i) Laboratory glassware such as conical flasks, beakers etc.
- (j) Fume cupboard.

3. Reagents

- (a) Chloroform, LR grade is suitable.
- (b) Isopropyl Alcohol.
- (c) Borax (Sodium tetraborate).
- (d) Hydrochloric Acid Standard Solution (0.5M). Add 42.5 mL of concentrated AR hydrochloric acid (density 1.19 g/mL) to 500 mL of isopropyl alcohol in a 1000 mL volumetric flask. Dilute to volume with water and mix. Standardize with borax.

CAUTION: Add the acid in small amounts, swirling the flask after each addition. Great care is always necessary when diluting concentrated acids, as heat generated by improper mixing may be sufficient to cause local boiling and result in the 'spitting' of hot acid droplets. The wearing of safety goggles or the equivalent is always necessary.

- (e) Isopropyl alcohol solution. Add 5 mL water to 95 mL isopropyl alcohol.

4. Standardisation of pH Meter

Follow the manufacturer's instructions and standardise at pH's of 4.0 and 7.0.

5. Procedure

- (a) Weigh approximately 0.5 g of sample accurately into a 250 mL beaker.
- (b) Add 90 mL of chloroform and 10 mL of isopropyl alcohol solution (3e) cover with a watch glass and boil for 1 minute on a hot plate. Cool to room temperature.

Note: Perform this step in the fume cupboard.

- (c) Insert the stirrer bar in the beaker and place on the magnetic stirrer. Adjust the pH electrodes and beaker so that they are just free off the bottom yet the stirrer bar does not hit the electrodes. Start the stirrer and adjust the speed so that there is vigorous stirring without spattering and the electrodes are covered with liquid.

- (d) Titrate with 0.5 M HCl using the millivolt scale. Record the millivolt reading at approximately 0.5 mL intervals. In the vicinity of the end point, when the millivolt increments become larger, record the millivolt readings every 0.1 mL.

6. Calculations

- (a) Calculate the first $(\Delta E/\Delta V)$ and the second $(\frac{\Delta^2 E}{\Delta V^2})$ derivatives and draw a plot of $\Delta^2 E/\Delta V^2$ as ordinate versus volume as abscissae. Read off the end point where the graph crosses the zero axis (see Vogel, 1961 p930). The procedure is illustrated in the table below, these being actual figures obtained in a titration.
- (b) From the end point titre, calculate the total amine value as follows:

$$\text{Total amine Value} = \frac{V \times M \times 56.1}{S} \text{ over}$$

Where

V = millilitres of HCl required for titration (from graph).

M = Molarity of HCl.

S = Sample mass in grams.

Report the total amine value to the nearest whole number.

EXAMPLE OF TITRATION FIGURES AND CALCULATIONS

TITRE VOL	mV	$\frac{\Delta E}{\Delta V}$	$\frac{\Delta^2 E}{\Delta V^2}$
.7	104		
1.0	81	-76.7	-20
1.3	52	-96.7	-53
1.6	7	-150	-10
1.7	-9	-160	-50
1.8	-30	-210	-90
1.9	-60	-300	-380
2.0	-128	-680	-1340
2.1	-330	-2020	1650
2.2	-367	-370	210
2.3	-383	-160	70
2.4	-392	-90	10
2.5	-400	-80	35
2.7	-409	-45	15
2.9	-415	-30	