

Method to determine free water-soluble acid in crude tall oil

Scope

This method describes a procedure for determining the free water soluble acid in crude tall oil by titration with alkali. A knowledge of the free water soluble acid is important in ensuring correct acidulation of tall oil soap. The presence of excess free acid in tall oil can cause corrosion of distillation equipment.

Apparatus

1. Separatory funnel, 250-mL.
2. Erlenmeyer flask, 250-mL.

Reagents

1. Toluene.
2. Distilled water.
3. Phenolphthalein indicator, 1%.
4. Potassium hydroxide, 0.1 N solution, standardized to + or - 0.001.

Procedure

1. Heat the sample at 80^o to 90^oC for at least 30 minutes. Stir vigorously to homogenize.
2. Weigh a 10 g sample into a 250-mL separatory funnel and cool to room temperature.
3. Add 50 mL of toluene to the funnel and shake to dissolve. Vent the funnel.
4. Add 20 mL of distilled water and shake vigorously. Vent, allow the layers to separate

and draw off the lower, aqueous layer into a 250-mL flask.

5. Repeat the water wash four times.
6. Combine the extracts in the 250-mL flask and add 1-2mL of phenolphthalein indicator solution.
7. Titrate with 0.1 N KOH to the first faint permanent pink color. Alternately, use an automatic titrator.

Calculation

$$\text{Water soluble acid as H}_2\text{SO}_4, \% = \frac{A \times N \times 4.9}{W}$$

where:

A = KOH, mL
 N = normality of KOH solution
 W = weight of sample (dry basis), g
 4.9 = equivalent weight of sulfuric acid/10
 This is used in the equation to express water soluble acid as sulfuric acid.