

Method to determine the saponification value of tall oil

Scope

This method is used to determine the total acid content, both free and combined, of tall oil. (Acid number only measures the free acid). The combined acids are primarily esters formed by reaction with the neutral components present in the original tall oil. The saponification value is therefore a measure of tall oil quality. It is determined by measuring the alkali required to saponify the combined acids and neutralize the free acids.

Apparatus

1. Erlenmeyer flask, 300-mL with S/T 24/40 neck and reflux condenser.
2. Pipet, 25-mL.
3. Automatic titrator (optional).

Reagents

1. Potassium hydroxide, 0.5 N solution in 3A etjyl alcohol, standardized to ± 0.005
2. Hydrochloric acid, 0.5 N solution, standardized to ± 0.005 .
3. Phenolphthalein indicator, 1% (visual titration only).

Procedure

1. Weigh 2 g of sample, to the nearest 0.01 g, into a 300-niL Erlenmeyer flask.
2. Using a pipet, add 25 n1L of 0.5 N ethanolic potassium hydroxide.
3. Reflux for 60 minutes.
4. Titrate between 60 and 70°C with 0.5 N hydrochloric acid using phenolphthalein indicator or an automatic titrator.
5. Run a blank in the same manner.

Calculation

$$\text{Saponification Value} = \frac{(A - B) \times N \times 56.1}{W}$$

where:

A =	H ₂ SO, for blank, mL
B =	H ₂ SO, for sample, mL
W =	weight of sample (dry basis), g
N =	normality H ₂ SO ₄ solution
56.1 =	equivalent weight of potassium hydroxide

Precision statement

Based on an ASTM round-robin study, the within laboratory (repeatability) standard deviation for this test is 0.8 and the between laboratory (reproducibility) standard deviation for this test is 1.4.

Reference

ASTM D464 "Saponification Number of Naval Stores Products Including Tall Oil and Other Related Products." ■