

Method of determining acid number of tall oil using a colorimetric end point

Scope

This method is used to determine the acid number of crude tall oil (CTO) and other related products using the colorimetric indicator technique for determining the equivalence points of the titration.

Use of a colorimetric indicator yields a less accurate value with lower precision compared to the potentiometric technique. Dark samples (Gardner ≥ 12) are difficult to analyze using the colorimetric end point.

The sample is dissolved in a mixture of isopropyl alcohol/toluene and titrated with standardized alcoholic potassium hydroxide to a colorimetric end point.

Apparatus

1. Buret, 50-mL with 0.1 mL divisions.
2. Stirrer, variable-speed with polytetrafluoroethylene (PTFE) coated magnetic stir bar or other type of mechanical stirrer.
3. Erlenmeyer flask, 250-mL.

Reagents

1. Standard alcoholic alkali (potassium hydroxide) solution, 0.5N - Purchase or prepare by dissolving 33 g of potassium hydroxide (pellets or sticks) in methanol or 39 mL of 45% KOH solution in a one liter bottle, and dilute to approximately one liter with the alcohol. Standardize to 0.001N by dissolving approximately 2.5 g of potassium acid phthalate (KHP) in 60 mL of water followed by the addition of 40 mL of isopropanol once the KHP has dissolved; 2.553 g of KHP will be neutralized by 25.0 mL of 0.5N KOH solution.

Protect the standardized solution against evaporation and absorption of carbon dioxide (CO₂) from the air. The solution should be standardized weekly. The standardization should use the same equipment and techniques as used in the actual acid number determination.

2. Methanol, reagent grade.
3. Isopropanol, reagent grade.
4. Toluene, reagent grade.
5. Potassium acid phthalate (KELP), primary standard grade.
6. Indicator solution, dissolve 0.1 g of phenolphthalein or thymol blue in 100 mL of isopropanol.

Procedure

1. Accurately weigh 3-5 g of sample to the nearest 0.001 g, and transfer it to a 250-mL flask. Add 25 mL of toluene and swirl to dissolve.
2. Add 75 mL of isopropyl alcohol and swirl to mix.
3. Add four drops of indicator solution. Add the stir bar. Start the stirrer and adjust the speed so that there is vigorous stirring without spattering.

NOTE 1: For a dark solution, additional indicator may be needed to detect the endpoint

NOTE 2: As the end point is approached, the addition rate of titrant should be reduced to more accurately determine the end point

4. Titrate with the standard alkali solution to the first permanent faint pink color when using phenolphthalein or a faint blue-green color using thymol blue.

PCTM 2 Method of determining acid number of tall oil using a colorimetric end point /2

5. Read the buret to 0.05 mL titrant. Calculation

Calculate the acid number of the sample, expressed as milligrams of KOH per gram of sample, as follows, and report the value to the nearest whole number:

$$\text{Acid number} = \frac{(A \times N \times 56.1)}{B}$$

where:

- A = volume of alkali solution required for titration of the specimen, mL
N = normality of the alkali solution
B = sample weight, g
56.1 = equivalent weight of KOH and is used in the equation to express the acidity based on an equivalent amount of milligrams of KOH.

Reference

ASTM D465 "Acid Number of Naval Stores Products Including Tall Oil and Other Related Products."