

Method of determining residual soap in crude tall oil

Scope

This method is used to determine the residual soap content of crude tall oil using either a potentiometric or pH end point.

The test is used to determine the residual soap content of crude tall oil and other related process samples. The potentiometric method yields a more accurate value, with *better* precision, than the pH method. A weighed amount of crude tall oil is dissolved in toluene and isopropyl alcohol, and the residual soap titrated electrometrically with standardized hydrochloric acid.

Reagents

1. Standard hydrochloric acid solution, 0.1 N - Purchase or prepare by filling a 2-L volumetric flask approximately three quarters full with distilled water. Using a 250-mL graduated cylinder, transfer 17 mL of concentrated hydrochloric acid to the volumetric flask and dilute to the line with distilled water. Mix well by inverting the stoppered flask several times. Pipet 25 mL of the HCl solution into a 250-mL beaker containing 50-75 mL of distilled water. Titrate with standardized 0.1 N KOH using the same type of end point detection to be used with the actual analysis. Use pH 7 as the end point when using a pH meter.
2. Isopropanol, reagent grade.
3. Toluene, reagent grade.
4. Buffer, pH 4, commercially available.

Sample Preparation

1. Accurately weigh 25 g of CTO to the nearest 0.001 g into a 250-mL beaker.
2. Add 25 mL of toluene and swirl to dissolve.
3. Add 75 mL of isopropanol to the beaker and swirl to mix.

Method A- Potentiometric Titration (referee)

Apparatus

1. Automatic potentiometric titrator.
2. Analytical balance, capable of reading ± 0.001 g.
3. Beaker, 250-mL.
4. Magnetic stirrer with stir bar.

Procedure

1. Add a magnetic stir bar to the beaker if necessary.
2. Set up the autotitrator with the proper operating parameters and hydrochloric acid titrant.
3. Place the sample on the titrator and start the titration.
4. After the titration is complete, record the % soap printed out by the instrument or calculate the soap or soap number as in Calculations.
5. After each titration, rinse the electrode first with alcohol followed by water and store in buffer solution.

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Method B- pH Meter

Apparatus

1. pH meter with glass electrodes.
2. Buret, 10-ml, with 0.1 mL divisions.
3. Analytical balance, capable of reading
4. Beaker, 250-mL.
5. Magnetic stirrer with stir bar.

used in the equation to express soap

milligrams of KOH.
□ ±0.001g.

Procedure

1. Standardize the pH meter using pH 4 buffer.
2. Add a magnetic stir bar to the beaker and place on stirrer.
3. Insert the buffered electrode into the sample.
4. Titrate the sample to a pH of 4.0 with 0.1 N HCL and record the amount of titrant used.
5. After each titration, rinse the electrode first with alcohol followed by water and store in buffer solution.

Calculations

1. Calculate the percent residual soap content of the CTO to the nearest 0.1 %.

$$\text{Residual soap, \%} = \frac{A \times N \times 32.4}{B}$$

where:

- A = volume of acid solution required for titration of the specimen, mL
N = normality of the acid solution
B = sample weight, g
32.4 = value used to calculate the residual soap as sodium abietate.

2. Calculate the residual soap number of the CTO to the nearest 0.1 unit.

$$\text{Residual soap number} = \frac{A \times N \times 56.1}{B}$$

where:

56.1 = the equivalent weight of KOH and is used in the equation to express soap based on an equivalent amount of mg of KOH