

# Method of determining acid number of tall oil using a potentiometric end point

## Scope

This method is used to determine the acid number of crude tall oil using the potentiometric technique for determining the equivalence point of the titration.

The test is used to determine the acid number of crude tall oil (CTO) and other related products. The potentiometric method yields a more accurate value with better precision compared to the colorimetric indicator method.

The sample is dissolved in an isopropyl alcohol/toluene solvent and titrated with a standardized alcoholic potassium hydroxide to a potentiometric equivalence point.

## Apparatus

1. Glass electrode pH meter. Use either standard or alkali-resistant electrodes for this test. An automatic potentiometric titrator may be used in place of a pH meter.
2. Buret, 50-mL with 0.1 mL divisions.
3. Stirrer, variable-speed with PTFE coated magnetic stir bar or other type mechanical stirrer.
4. Beaker, 400-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 methyl alcohol or 39 mL of 45% KOH solution in a one liter bottle, and dilute to approximately one liter with the alcohol. Standardize to  $\pm 0.001N$  by dissolving approximately 2.5 g of potassium

acid phthalate (KHP) in 60 mL of water followed by the addition of 40 mL of isopropyl alcohol 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 ( $CO_2$ ) from the air. The solution should be standardized weekly potentiometrically. 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 (KHP), primary standard grade.
6. Buffer, pH 10, commercially available.

## Procedure

1. Accurately weigh 3-5 g of sample to the nearest 0.001 g, and transfer it to a 400-mL beaker. Add 25 mL of toluene and swirl to dissolve.
2. Add 75 mL of isopropanol and swirl to mix.
3. Adjust the beaker so the lower half of each electrode of the pH meter is immersed in the solution. Add the stir bar. Start the stirrer and adjust the speed so that there is vigorous stirring without spattering.

NOTE 1: If an autotitrator is employed, follow the manufacturer's operating instructions.

NOTE 2- Glass *electrodes* read to dehydrate in nearly anhydrous solvent medium. Condition the electrode in water between tests and check with known pH buffers frequently.