Content of tall oil in tall oil soap

Scope

This test method covers the determination of the quantity of crude tall oil (CTO) which may be obtained from a representative sample of tall oil soap.

This method is useful in predicting the yield of crude tall oil which may be obtained in commercial acidulation of tall oil soap as obtained from storage tanks, tank cars or process equipment.

Care must be taken to be sure that the sample being analyzed is representative of the material in the vessel, tank car or tank truck. Since some black liquor may separate from the soap in the sample container, the material should be homogenized just before removing an aliquot for this analysis.

Safety

Ethyl ether is extremely flammable and must be kept from sources of ignition. Perform all work in a fume hood.

Apparatus

1. Separatory funnels, 1000 mL and 500 mL.
2. Beaker, 250 mL for weighing soap sample.
3. Beaker, 400 mL.
4. Beaker, 600 mL.
5. Forced draft oven, maintained at 110 to 120°C.
6. pH indicator paper.

Reagents

1. Ethyl ether, reagent grade.
2. Sulfuric acid, 30% solution.
3. Sodium sulfate, 10 to 15% solution.

Procedure

1. Transfer approximately 50 g of tall oil soap into a 1000 mL separatory funnel. An efficient transfer can be made using portions of the 10-15% sodium sulfate solution.

2. Add 17 mL of 30% sulfuric acid solution, and shake the contents vigorously for 2-3 minutes, venting pressure at intervals as necessary.

3. Add 100 mL of ethyl ether and shake moderately until all of the soap has been reacted. Some lignin will be present but it will be indistinguishable from the soap. The pH of the aqueous layer should be between 2 and 3.

4. Allow the ether layer to separate, then draw off the aqueous layer into a 600 mL beaker. Leave the ether and lignin layers in the separatory funnel. Pour the ether extract from the top of the separatory funnel into a second 500 mL separatory funnel, taking care to exclude as much lignin as possible.

5. Add the aqueous layer from the 600 mL beaker back into the lignin layer remaining in the first separatory funnel. Extract with 3 more 100 mL portions of ether, adding each ether extract into the second separatory funnel. Wash the combined ether extracts with four (4) 50 mL portions of distilled water. The aqueous layers may be retained to determine the lignin and fiber content.

6. After washing the combined ether extract four times, incrementally add the extract into a tared 400 mL beaker containing a boiling stone, and evaporate to dryness on a steam bath. Rinse the separatory funnel with 30 mL of ether, add the ether rinse to the beaker, and evaporate to dryness.

7. After evaporating the ether from the combined extracts to dryness, dry the remaining extracted crude tall oil residue in the beaker in a 110 - 120°C oven for one hour, or as required to obtain a constant weight.

Calculation

Calculate the percent crude tall oil as follows:

$$Crude\,Tall\,Oil, \% = \frac{A}{B} \, x \, 100$$

where:

A   =   grams of extracted crude tall oil
B   =   sample weight of soap skimmings, g

Report

Report the crude tall oil content in the tall oil soap to the nearest 1.0%.

Reference

TAPPI Method T 635 om.

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