

# Recovery of tall oil from black liquor soap skimmings

## Scope

This method describes a laboratory procedure for conversion of black liquor soap skimmings to crude tall oil.

The procedure utilizes acidulation of the soap using sulfuric acid, to produce the crude tall oil and an aqueous sodium sulfate brine layer. The crude tall oil is washed with water to remove any residual brine or acid.

The resultant product crude tall oil is suitable for characterization by other chemical analyses as desired.

## Safety

Procedure steps 4 - 8 should be conducted in an appropriate fume hood, since toxic and noxious hydrogen sulfide may be released from the acidification of residual sulfide present in black liquor and soap skimmings.

## Apparatus

1. Top-loading balance, 0.01 g precision.
2. Hot plate, with suitable magnetic or mechanical stirrer.
3. Large oven maintained at 74-85 °C.

## Reagents

1. Sodium sulfate, anhydrous.
2. 79% Sulfuric acid (60° Baumé), commercial grade.
3. Methyl orange indicator, 1 % solution.

## Procedure

1. Place 300 mL of hot water into a 4liter beaker.
2. While agitating and heating, add 60 g of Na<sub>2</sub>SO<sub>4</sub>, (to facilitate the subsequent phase separation).
3. Add 25 mL of H<sub>2</sub>SO<sub>4</sub> (60° Baumé) and bring to a boil.
4. Decant any free black liquor present in the soap sample, and add 400 g of soap skimmings, in portions while maintaining agitation.
5. Again bring the solution to a gentle boil and hold for five minutes.
6. Remove the beaker from the heat and allow the brine to separate.  
Draw off a small portion of the brine and check the pH. The desired pH is between 3.0 and 3.5. If the pH is high, add Na<sub>2</sub>SO<sub>4</sub> and if the pH is low, add soap skimmings.
8. Once the correct pH range is obtained, allow the solution to gently boil for 30 minutes with agitation.  
Pour the mixture into a 1000 mL separatory funnel, and place in the oven at 74-85°C for approximately one hour to obtain separation of oil, lignin, and brine. Drain and discard the brine and lignin layer.
10. Wash the oil by adding 250 mL of water to the funnel, invert and mix moderately several times. Avoid vigorous mixing that may result in an emulsion. Then return the funnel to the oven to allow the aqueous phase to separate, as in Step 9. Repeat this washing step until the aqueous phase is neutral (pH 5.5 to 7.0) to methyl orange.
11. Remove the funnel from the oven, and discard the aqueous layer.
12. Retain the top layer, which is the crude tall oil.