

## Method to determine lignin, fiber, and foreign matter in tall oil soap

### Scope

This method is used to determine lignin as well as fiber and foreign matter in tall oil soap. The soap sample is acidulated to convert soap to tall oil. The mixture is filtered and the tall oil dissolved in hexane. The undissolved solids which contain lignin, fiber, and foreign matter are washed with methanol to remove lignin. The material remaining on the filter paper is dried and weighed to determine fiber and foreign material. Water is used to precipitate the lignin from the methanol filtrate. The lignin is filtered and weighed.

### Apparatus

1. Beaker, 400-mL.
2. Filter paper, No. 40 Whatman, 2 pieces.
3. Buchner funnel with 1-liter side arm flask.
4. Balance sensitive to 0.001 g.
5. Graduated cylinder, 100-ml.

### Reagents

1. Hexane, reagent grade.
2. Methanol, absolute.
3. Sulfuric acid, 30% solution.

### Procedure

1. Weigh 200 g of a representative soap to the nearest 0.001 g. Record this weight as W.
2. Convert the soap to oil using PCTM 6.
3. Tare a piece of filter paper (to 0.001 g) and record this weight as D. This value will be used to calculate % fiber and foreign matter.
4. With vacuum, filter the tall oil and brine using the Buchner funnel. Filter the oil first (thinning the oil with hexane will save time) then the brine.

5. Wash the filter paper with hexane to remove residual tall oil. The filter now contains lignin, fiber, and other foreign matter.
6. Discard the filtrate.
7. Wash the filter with methanol until all of the lignin has dissolved and the filtrate is clear. Collect the methanol wash. The methanol must be dry to dissolve the lignin.
8. Dry the filter paper to a constant weight in an oven at 105 °C. Record this weight to the nearest 0.001 g, as C. This value will be used to determine % fiber and foreign matter.
9. To the methanol, add twice as much water as methanol to precipitate the lignin.
10. Tare a second piece of filter paper to the nearest 0.001 g, and record this weight as B. This value will be used to calculate % lignin.
11. Filter the precipitated lignin through the tared filter paper.
12. Dry the filter paper to a constant weight at 105 °C measured to the nearest 0.001 g. Record this weight as A. This value will be used to determine % lignin.

### Calculations

$$\text{Lignin, \%} = \frac{(A - B)}{W} \times 100$$