# Moisture content of tall oil by azeotropic distillation

## Scope

A sample is dissolved in toluene or xylene, then the azeotrope of the solvent and water is distilled and the water is collected. The volume of the separated water is used to calculate the percent moisture.

## Apparatus

1.   Flask, 0.5- or 1-liter short-neck, round-bottom or Erlenmeyer flask, depending upon the weight of the sample used. The larger size is preferred to avoid foaming.
2.   Hot plate or heating mantle.
3.   Reflux condenser, condenser connected to the flask and discharging into a trap.
4.   Trap, 5-mL trap, subdivided into 0.1 mL, divisions, with each I mL line numbered.

## Reagents

Toluene or xylene - reagent grade, moisture free.

## Procedure

1.   Weigh 100 grams of tall oil into the flask Add a few boiling stones. Fit the flask with a moisture trap and a reflux condenser. Add 150 mL of toluene or xylene to the flask and fill the moisture trap with solvent.
2.   Heat gently until the solvent begins to reflex. Continue vigorous refluxing for 2 hours. Wash down the condenser with 5 mL portions of solvent from time to time during the reflux period and with four 5 mL, portions of solvent at the end. A drop of water remaining on the condenser may be dislodged with a fine wire or a stirring rod wet with methanol. Record the volume of water in the trap.

## Calculation

$$Moisture, \% = \frac{V}{W} \; x \; 100$$

where:

$V$ =    Volume of water, n1L

$W$ =    Weight of sample, g

## Alternate methods

PCTM 4B, PCTM 4C

## Reference

ASTM D803 "Methods for Testing Tall Oil."