

Method to determine insolubles in crude tall oil

Scope

This method covers the determination of the amount of insoluble matter in crude tall oil (CTO). Crude tall oil is soluble in numerous organic solvents whereas most common contaminants are not.

Apparatus

1. Beaker, 800-mL.
2. Magnetic stirring hot plate with polytetrafluoroethylene-coated stir bar.
3. Filter paper, Whatman No. 40.
4. Two-piece filter apparatus appropriate to hold the filter without leaking.
5. Analytical balance, capable of weighing 0.0001 g.
6. Laboratory tweezers.
7. Forced draft oven, maintained at 105 to 110°C.
8. Desiccator.

Reagents

Toluene, Hexane, Mineral Spirits, or other good solvent for tall oil as defined by the customer and supplier.

Procedure

1. Wash a filter with solvent, dry and store in a desiccator until ready to use.
2. Place a pre-weighed filter in the filter apparatus and secure it to prevent leakage. Record the weight (A), to the nearest 0.0001 g.

NOTE 1: Always use tweezers when handling the filter.

Weigh 100 ± 0.1 g of sample into an 800-mL beaker. Record the weight (B). Add 150 mL of solvent. Place a PTFE-coated magnetic stir bar

into the beaker, and place the beaker on a hot plate. Heat and stir the material until it is completely dissolved. Do not boil the solvent.

4. Immediately pour the solution through the filter. Rinse the beaker and filter apparatus three times with additional hot solvent.
5. Disassemble the filter apparatus, remove the filter, and place it, contaminated side up, in a forced draft oven. Dry the filter to constant weight at 105 to 110°C (1 hr. is usually sufficient), cool in a desiccator, and weigh. Record the weight of the dry contaminated filter to the nearest 0.0001 g (C).

Calculation

$$\text{Insolubles, \%} = \frac{C-A}{B} \times 100$$

where:

- A = weight of clean, dry filter paper, g
B = weight of sample, g
C = weight of filter paper and insolubles, g

Report

Report the percent insoluble matter to the nearest 0.01%.

References

TAPPI Method T 621 cm
ASTM D269 "Insoluble Matter in Rosin and Rosin Derivatives." ■