

Method to determine ash content of tall oil soap

Scope

This procedure is used to determine the residue after ignition at red heat.

Apparatus

1. Crucible - porcelain, silica or platinum, 70-mL capacity.
2. Desiccator.
3. Bunsen burner.
4. Muffle furnace.
5. Oven, general purpose, laboratory.

Reagents

Anhydrous ethanol or 3A ethanol (optional).

Procedure

1. Heat a clean crucible at $750^{\circ} \pm 25^{\circ} \text{C}$ in a muffle furnace for 20 minutes.

NOTE 1: A platinum crucible is preferred if the ash is to be analyzed later for metals.

2. Place the crucible in a desiccator and allow to cool to ambient temperature.
3. Weigh the crucible to the nearest 0.001 g. Record the Weight
4. Place approximately 20 g of the sample in the crucible and weigh to the nearest 0.001 g. Record this weight.
5. Pre-dry the sample overnight at 110°C in a drying oven.

NOTE 2: If a more rapid determination is desired, do not pre-dry. Instead add 1-2 mL of anhydrous ethanol (3A ethanol may also be used). Then using a bunsen burner, gently heat the crucible until the sample is completely dry and carbonized if the sample ignites, allow to burn until the flame goes out.

6. Put the crucible and sample back into the muffle furnace for 30 minutes (at 750°C).
7. Return the crucible to the desiccator and allow to come to ambient temperature. Weigh the crucible and residue to the nearest 0.001 g. Record this weight.

Calculation

$$Asb, \% = \frac{(A-B)}{C-B} \times 100$$

where:

A	=	Weight of crucible plus residue, g
B	=	Weight of crucible, g
C	=	Weight of crucible plus sample, g

Reference

ASTM D803 "Methods for Testing Tall Oil." ■