

EXTRANEOUS MATERIALS

PRINCIPLE

The syrup sample is diluted with hot purified water and the insoluble material is collected by vacuum filtration on a dried and tared filter paper. After drying and weighing, the insoluble material is identified by microscopic examination.

SCOPE

The method can be applied to syrups, crude and refined sugars and other water soluble materials.

SPECIAL APPARATUS

1. Filter Paper: Whatman No. 1 or Schleicher and Schuell No. 597, 5.5 cm diameter, is recommended.
2. Funnel: A sintered plate Buchner funnel constructed so that the filter paper lies flat and covers the entire filtering area is necessary. The 2.5 inch diameter, coarse porosity, stainless steel funnel supplied by Scientific Glass Apparatus Company, Inc. (Cat. No. F-8125, size BBM) is satisfactory if the inside rim is turned down so that it is flush with the sintered plate. The funnel is attached to a large filter flask connected to a vacuum supply (Note 1).
3. Microscope: A wide field, binocular type instrument, with inclined oculars providing magnification in the range of 15 to 45 X, is recommended.

PROCEDURE

Center a 5.5 cm filter paper (Note 2) on the sintered plate of the funnel, apply vacuum, and wash the paper with portions of hot (about 80 °C) purified water totaling about 200 mL. Place paper in previously dried and tared aluminum or glass dish equipped with cover, and dry 1 hour in vacuum oven at 100 °C. Remove from oven, cover dish quickly, cool in desiccator, and weigh.

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Weigh 500 g of syrup into a 2 L beaker, add 1 L of hot (about 80 °C) purified water, and stir until dissolution is complete. Center the prepared filter paper on the sintered plate of the funnel, moisten with water, and apply vacuum.

Filter the solution immediately, rinsing all residue into the funnel with water, and wash the residue and filter paper thoroughly with portions of hot (about 80 °C) purified water totaling about 200 mL (Note 3).

Place the filter paper and residue in dish used originally, and dry 1 hour in a vacuum oven at 100 °C. Remove from oven, cover dish quickly, cool in desiccator, and weigh.

After determining residue weight, carefully transfer paper with residue to stage of wide-field microscope. Moisten paper with a few drops of water to avoid residue loss and facilitate viewing. Using magnification suitable for identification of residue particles, examine entire paper and note significant residue particles (Note 4).

CALCULATION

$$\text{Extraneous Material, ppm} = \frac{\text{Residue Wt. (g)} \times 1,000,000}{\text{Sample Wt. (g)}}$$

Identify and report nature of extraneous material.

NOTES AND PRECAUTIONS

1. A coarse porosity, fritted glass, Buchner form funnel, having a capacity of 150 mL and a nominal disc diameter of 60 mm, usually operates satisfactorily. The actual disc diameter is about 57 mm; the outer edge of the glass frit is fused and insoluble material does not escape around the edge of a centered 55 mm filter paper. The outer edges of the glass frit can be painted with two or three coats of an epoxy or polyester resin if necessary.
2. The recommended filter papers are white which facilitates microscopic examination of colored extraneous material. Some residues are white, and

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are difficult to distinguish from the paper background during visual examination and identification.

3. Perform analysis in a clean atmosphere to prevent contamination by air-borne materials.

3. Extraneous material is defined here as non-syrup or foreign material. It may consist of contamination from processing equipment or materials, or air-borne contamination. Long experience facilitates visual identification of most residue particles recovered by this procedure.

METHOD HISTORY

Corn Syrup, Extraneous Materials (E-27), Date of Acceptance 11-07-1966, Revised 3-31-1992.