

EXTRANEOUS MATERIALS

PRINCIPLE

The product sample is diluted with deionized or distilled 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

This method applies to citric acid final product samples, both liquids and solids. Use a gravimetric method in the case of excessive matter.

SPECIAL APPARATUS

1. Filter: 0.8 micron membrane filter or 934 AH, 1.5 micron with 9.0 cm diameter; Whatman No. 1 or Schleicher and Schuell No. 597, 5.5 cm diameter; or equivalent.
2. Funnel: A sintered plate Buchner funnel, VWR Cat No. 30310-109 or equivalent, constructed so that the filter paper lies flat and covers the entire filtering area is necessary. A 2.5 inch diameter, coarse porosity, stainless steel funnel 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. Stereoscope: A wide field, binocular type instrument, with inclined oculars providing magnification in the range of 15 to 45 x, is recommended.
4. Vacuum oven: Operate at 70°C, and at a pressure not to exceed 25 torr.

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) 0.2 micron-filtered

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DI 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.

Weigh 500 g of product in 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) deionized or distilled water totaling about 200 ml (Note 3).

Place the filter paper and residue in the dish used originally and dry for 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 the paper with residue to the stage of a wide-field stereoscope. Moisten the paper with a few drops of water to avoid loss and facilitate viewing. Using 25x magnification, examine entire paper and note residue particles (Note 4).

CALCULATIONS

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

Identify and report the nature of any extraneous material.

NOTES AND PRECAUTIONS

1. A coarse porosity, fritted glass, Buchner 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.

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2. The recommended filter papers are white which facilitates microscopic examination of colored extraneous material. Some residues are white and are difficult to distinguish from the paper background during visual examination and identification.
3. Perform analysis in a clean atmosphere to prevent contamination by airborne materials.
4. Extraneous material is here defined as non-product or foreign material. It may consist of contamination from processing equipment or materials, or airborne contamination. Long experience facilitates visual identification of most residue particles isolated by this procedure.

REFERENCE

Society of Soft Drink Technologists, Sweetener Analysis, Procedure 9.0, Extraneous Materials, High Fructose Corn Syrup, Oct. 1994.

METHOD HISTORY

Lactic Acid, Extraneous Materials (L-4), Date of Acceptance 9-08-2006.