SULFATE

PRINCIPLE

Soluble sulfates are precipitated directly form a diluted and acidified aqueous solution of the sample by addition of barium chloride. The insoluble barium sulfate is removed by filtration, washed, ignited and weighed.

SCOPE

This procedure is applicable to corn syrup and finished corn sugars.

SAFETY

Analysts should be familiar with the use and disposal of acids. Adequate personal protective equipment should be worn. All operations of transfer of concentrated acid to water and of evaporation should be carried out under a fume hood.

SPECIAL APPARATUS

1. Furnace: A muffle furnace equipped with a pyrometer and capable of operating at temperatures up to 525 °C is recommended.

2. Porcelain filter crucibles: 25 mL, porosity No. 4 (5-10 μm) suitable for the retention of barium sulfate and available from most scientific suppliers.

REAGENTS

1. Barium Chloride Solution, 10%: Dissolve 100 g of barium chloride dihydrate ($BaC1_2 \bullet 2H_2O$) in distilled water, dilute to 1 L volume and mix thoroughly.

2. Hydrochloric Acid, Concentrated (37% HCl, sp g 1.19).

PROCEDURE

Weigh accurately about 50 g of sample into a 400 mL beaker, dissolve in approximately 250 mL of distilled water and add 3 mL of concentrated hydrochloric acid. Gravity filter solution through a good quality filter paper to remove any insoluble solids. Rinse beaker, filter paper and funnel with a small

SULFATE — continued

volume of distilled water and collect washings together with filtrate in a 600 mL beaker. Heat the solution to incipient boiling and add slowly 10 mL of barium chloride solution while stirring the solution vigorously. Boil solution gently for 5 minutes, remove from heat and allow precipitate to settle 2 hours or preferably overnight. Filter the solution through a filter crucible, previously ignited 1 hour at 525 °C, and tared, collecting the precipitate quantitatively. Treat filtrate with barium chloride solution to check completeness of precipitation. Wash precipitate thoroughly with hot water (about 50 mL). Remove excess water from crucible contents by drying in warm air oven. Place crucible in muffle furnace at about 525 °C, ignite to constant weight (2 hours usually sufficient), cool and weigh.

CALCULATION

$$\%\ \text{Sulfate (as is)} = \frac{\text{Residue Wt. (g)} \times 0.412 \times 100}{\text{Sample Wt. (g)}}$$

REFERENCE

G.H. Jeffrey et. al., Ed., *Vogel's Textbook of Quantitative Chemical Analysis, Fifth Edition* (1989), *Longman Scientific Technical* (John Wiley and Sons), 11.72, pp. 490-493.

METHOD HISTORY

Corn Syrup, Sulfate (E-64), Date of Acceptance 4-05-1954, Revised 10-23-2001.

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