

HEAVY METALS

PRINCIPLE

Heavy metals are here defined as those metallic impurities that are colored by hydrogen sulfide under the conditions of this test. They are determined by visual comparison with standard solutions and expressed as lead (Note 1).

SCOPE

This method is applicable to corn syrups and corn sugars.

SPECIAL APPARATUS

Nessler Color Comparison Tubes: 50 mL capacity, tall form

REAGENTS

1. Acetic Acid Solution, 6%: Dilute 60 mL of reagent grade glacial acetic acid to 1 L with purified water and mix thoroughly.
2. Hydrogen Sulfide Solution, Saturated: Pass hydrogen sulfide gas through cold water until saturated. The saturated solution should produce, at once, a copious precipitate of sulfur when added to an equal volume of 10% ferric chloride solution.
3. Standard Lead Solution: Prepare a stock solution by dissolving 0.1598 g of reagent grade lead nitrate ($\text{Pb}(\text{NO}_3)_2$) in 100 mL of purified water containing 1 ml of nitric acid. Dilute to 1 L with purified water and mix thoroughly. As required, dilute 10.0 mL of this stock solution to 100 mL with purified water and mix. One mL of this standard solution contains 0.01 mg of lead (Note 2).

PROCEDURE

Weigh accurately about 5 g of sample and transfer with a minimum quantity of purified water to a 50 mL Nessler tube (Note 3). Pipet 0.0, 1.0, 2.0, 3.0, 4.0 and

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5.0 mL of standard lead solution into 6 separate 50 mL Nessler tubes. From this point treat all tubes in the same manner.

Add 2 mL of 6% acetic acid, dilute to 25 mL with purified water and mix. Add 10 mL of saturated hydrogen sulfide solution, mix and allow to stand 10 minutes. Compare color in sample tube with that in standard tubes by viewing downward over a white surface.

Determine the volume of standard lead solution (to nearest 0.5 mL) required to obtain a color match.

CALCULATION

Heavy Metals, ppm (as is, expressed as lead)

$$= \frac{\text{mL Std. Pb Soln.} \times 0.00001 \times 1,000,000}{\text{Sample Wt. (g)}}$$

NOTES AND PRECAUTIONS

1. Lead, bismuth, copper, silver and tin are the principal contributors to the heavy metals value obtained by this colorimetric procedure. Other metals in groups I and II (qualitative analytical classification) do not contribute significantly because their sulfides are white or lightly colored, or because the sulfide salt is not precipitated as is the case with mercury.
2. Dilute standard lead solution must be freshly prepared. Stock solution may be stored in glass free from soluble lead salts.
3. Sample transfer is accomplished readily by dissolving sample in weighing dish in about 10 mL of purified water followed by pouring dissolved sample into Nessler tube and rinsing dish with another 5 mL portion of purified water.