IRON (TPTZ)

PRINCIPLE

Iron is reduced quantitatively to iron (II) in the presence of hydroxylamine. The reagent reacts with iron, after reduction to iron (II), to produce an intense violet-colored complex highly specific for iron (II). The absorbance of the complex is measured spectrophotometrically at 593 nm and is reacted with 2,4,6-tripyridyl-s-triazine (TPTZ) to produce an intense violet colored complex. Standard curves show excellent day-to-day reproducibility and colors are stable for at least 24 hrs.

SCOPE

The method is applicable to corn syrup with or without a prior ashing step. The method is generally applicable to organic and inorganic materials following an appropriate ashing step where required to provide a sample containing solubilized iron (Note 1).

SPECIAL APPARATUS

Double Beam Spectrophotometer: An instrument capable of accurate absorbance measurements at 593 nm and equipped with a matched pair of 1 cm cuvettes. It should be equipped with matching 1 and 5 cm cuvettes.

REAGENTS

1. Stock Iron Solution, 100 µg/mL iron: Dissolve 0.702 g of reagent grade ferrous ammonium sulfate hexahydrate (Fe(NH₄)₂(SO₄)₂•6H₂O) in 50 mL of purified water, add 20 mL of 1:3 sulfuric acid and dilute to 1 L with purified water.

2. Standard Iron Solution, 5 µg/mL iron: Pipet 25 mL of stock iron solution into a 500 mL volumetric flask and dilute to volume with purified water.

3. Hydroxylamine Hydrochloride Solution, 10%: Dissolve 50 g of reagent grade hydroxylamine hydrochloride (NH₂OH•HCl) in 300 mL of purified water.
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water, transfer to a 500 mL volumetric flask and dilute to volume with purified water (Note 2).

4. Sodium Acetate-Acetic Acid Buffer: Dissolve 164 g of reagent grade anhydrous sodium acetate (NaC₂H₃O₂) in 600 mL purified water, add 115 mL glacial acetic acid (99.7% CH₃COOH; sp g 1.06), and dilute to 1 L with purified water.

5. Reagent: Dissolve 0.312 g TPTZ in 1 mL of concentrated hydrochloric acid (37% HCl; sp g 1.19) and dilute to 1 L with purified water.

PROCEDURE

Direct: For samples containing less than 1 ppm iron, weigh a 10.0 g sample, dissolve in warm purified water and transfer quantitatively to a 100 mL volumetric flask. Add in order, 4 mL hydroxylamine hydrochloride solution, 20 mL buffer and 10 mL TPTZ reagent. Dilute to volume with purified water. Prepare standards by pipetting 1.0, 2.0 and 5.0 mL standard iron solution into separate 100 mL volumetric flasks; add reagents and dilute to volume with purified water. Determine the transmittance of standards and samples vs. a reagent blank in 5 cm cuvettes at 593 nm (Note 3).

For samples containing 1-5 ppm iron, weigh a sample containing 20-100 µg iron, transfer quantitatively to a 100 mL volumetric flask and add 4 mL hydroxylamine hydrochloride solution, 20 mL buffer and 10 mL TPTZ reagent. Dilute to volume with purified water. Prepare standards by pipetting 5, 10 and 20 mL of standard iron solution into separate 100 mL volumetric flasks. Add reagents and dilute to volume with purified water. Determine the transmittance of standards and samples vs. a reagent blank in 1 cm absorption cells at 593 nm.

A Lansing: If insoluble iron is present or suspected, an ashing step is needed.

Weigh accurately a sample containing 5-100 mL µg iron (not more than 10 g) into a tared platinum or silica dish. Add 5 mL of 1:3 sulfuric acid and heat on a hot plate until thoroughly carbonized. Place in a muffle furnace at 525 °C and heat for 2 hrs. or until ash is practically carbon free. Cool, treat residue with 5 mL of 1:1 hydrochloric acid and evaporate to incipient dryness on a steam bath. Treat
residue with 2 mL of 1:1 hydrochloric acid and 10 mL purified water, warm on a steam bath to assist dissolution. If necessary, gravity filter to remove any insoluble residue, collect filtrate in a 100 mL volumetric flask, and wash residue with purified water to ensure quantitative transfer. Add reagents as described in the Direct procedure and determine the transmittance of sample vs. reagent blank as described above.

**CALCULATIONS**

Prepare a standard curve by plotting log % T vs. iron content (µg/100 mL) for the standard solutions.

Determine iron content of the sample solution by reference to the appropriate standardization curve.

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\text{Iron, ppm (as is)} = \frac{\mu g \text{ Iron from Graph}}{\text{Sample Wt., g}}
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\text{Iron, ppm (d.b.)} = \frac{(\text{Iron, ppm as is})(100)}{\text{Sample Dry Substance, %}}
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**NOTES AND PRECAUTIONS**


2. If necessary, iron can be removed from this and other reagents by extraction of the iron-TPTZ complex with nitrobenzene (caution) as described in the reference in Note 1.