Determination of Iron in Water

Introduction

Use spectrophotometric methods to determine the concentration of iron in a water sample.

Concepts

- Spectrophotometric analysis
- Water quality testing

Materials

1,10-phenanthroline, $C_{12}H_8N_2 \cdot H_2O$, 0.1 g
Acetic acid, glacial, CH_3CO_2H, 9 mL
Hydroxylamine hydrochloride, $H_2NOH \cdot HCl$, 0.5 g
Iron(II) ammonium sulfate hexahydrate, $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$, 35.1 mg
Sodium acetate, $CH_3CO_2Na \cdot 3H_2O$, 10 g
Sulfuric acid, H_2SO_4, 1.0 mL
Buret, 50 mL
Graduated cylinder, 10-mL
Spectrophotometer with cuvets
Volumetric flask, 100-mL, 6
Volumetric flask, 250-mL

Safety Precautions

The sodium acetate buffer, hydroxylamine hydrochloride solution, glacial acetic acid, and 1,10-phenanthroline are toxic by ingestion. Sulfuric acid and glacial acetic acid are severely corrosive to the eyes, skin, and other tissue. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Please review current Material Safety Data Sheets for additional safety, handling, and disposal information.

Preparation

1. Prepare a stock iron solution by dissolving 35.1 mg of iron(II) ammonium sulfate hexahydrate in 100 mL of distilled water in a 250-mL volumetric flask. Add 1.0 mL of concentrated sulfuric acid, H_2SO_4, and fill to the mark with distilled water. Unknown solutions can be made by diluting the stock iron solution. Make sure to keep track of the concentration.
2. Prepare a sodium acetate buffer by dissolving 10 g of sodium acetate in 50 mL of distilled water in a 100-mL volumetric flask. Slowly add 9 mL of glacial acetic acid and dilute to 100 mL with distilled water.
3. Prepare a hydroxylamine hydrochloride solution by dissolving 0.5 grams of hydroxylamine hydrochloride in 50 mL of distilled water.
4. Prepare a 1,10-phenanthroline solution by dissolving 0.1 g of 1,10-phenanthroline in 30 mL of distilled water with the aid of stirring and gentle heating. Cool and dilute to 50 mL. Store in a dark place.

Procedure

Preparation of Standard Iron Solutions and Calibration Curve.

1. Using a buret, transfer 2.00, 4.00, 8.00, 10.00 and 12.00 mL of the stock iron solution into separate 100-mL volumetric flasks. Fill each flask about half full with deionized or distilled water.
2. Using a graduated cylinder, add 10.0 mL of the sodium acetate buffer to each flask.
3. Using a pipet or a graduated cylinder, measure and add 2.0 mL of the hydroxylamine hydrochloride solution to each flask and mix.
4. Finally, add 5.0 mL of the 1,10-phenanthroline solution to each flask. Fill each flask to the 100-mL mark with distilled or deionized water and mix. Calculate the concentration of iron in mg/100mL.
5. To prepare a blank, add 10.0 mL of buffer, 2.0 mL of hydroxylamine hydrochloride solution, and 5.0 mL of 1,10-phenanthroline solution to a 100-mL flask. Dilute to the mark with distilled or deionized water and mix. This is your blank solution.

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6. You now have five standard iron solutions and one blank. Transfer portions of the solutions to the spectrophotometer test tubes. Using the blank, zero the instrument at a wavelength of 508 nm. Take readings for each of the standard solutions.
7. Graph a calibration curve of Absorbance vs. Concentration of iron knowing that the standard iron solution contains 2.0 mg Fe/100-mL. Therefore, you know that the first standard solution contains 0.04 mg Fe/100 mL of total solution.

Analysis of Unknown

1. Obtain 50.0 mL of an unknown iron solution or a sample of tap water. Place in a 100 mL volumetric flask and treat as above (steps 2 through 5). Transfer a portion of the solution to a spectrophotometer test tube.
2. Read the absorbance.
3. Locate the absorbance of the unknown solution on the calibration curve and determine the concentration of iron in that solution. The concentration of the sample is one-half the concentration of the unknown.
4. Analyze other unknowns if time allows.

Disposal

Please consult your current Flinn Scientific Catalog/Reference Manual for general guidelines and specific procedures, and review all federal, state and local regulations that may apply, before proceeding. All solutions and reagent wastes must be collected and transferred to a licensed hazardous waste disposal company for proper treatment.

Connecting to the National Standards

This laboratory activity relates to the following National Science Education Standards (1996):

Unifying Concepts and Processes: Grades K–12
Systems, order, and organization
Evidence, models, and explanation
Constancy, change, and measurement

Content Standards: Grades 5–8
Content Standard A: Science as Inquiry
Content Standard B: Physical Science, properties and changes of properties in matter
Content Standard E: Science and Technology
Content Standard F: Science in Personal and Social Perspectives, personal health; populations, resources, and environments; natural hazard

Content Standards: Grades 9–12
Content Standard A: Science as Inquiry
Content Standard B: Physical Science, structure and properties of matter, chemical reactions
Content Standard E: Science and Technology
Content Standard F: Science in Personal and Social Perspectives, personal and community health, natural resources, environmental quality, natural and human-induced hazards, science and technology in local, national, and global challenges

Discussion

The EPA recommends that the concentration of iron in drinking water should not exceed 0.30 mg/liter. Since the daily nutritional requirement of iron is 1 to 2 mg, the standard is for aesthetic reasons rather than toxicity. It should be pointed out, however, that iron concentrations of above 1.0 mg/liter are detrimental to many freshwater fish, especially trout.

This method is best suited for detecting small amounts of iron in water (0.001 to 0.05 mg). Iron(III) iron must be reduced to the iron(II) state using hydroxylamine hydrochloride. The determination depends upon the intense red complex which the iron(II) ion forms with 1,10-phenanthroline. Three molecules of 1,10-phenanthroline chelate each atom of iron. The equation for the reduction of the iron(III) ion to the iron(II) ion is:

$$4Fe^{3+} + 2NH_3O \rightarrow 2N_2O + 4Fe^{2+} + 6H^+$$

Acknowledgment

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Reference

Flinn Scientific Spectrophotometer Laboratory Manual; Flinn Scientific: Batavia, IL, 1994; pp 55–60.

Materials for *Determination of Iron in Water* are available from Flinn Scientific, Inc.

Catalog No.	Description
F0013	Iron(II) Ammonium Sulfate, 100 g
S0036	Sodium Acetate, 100 g
A0177	Acetic Acid, Glacial, 100 mL
H0052	Hydroxylamine Hydrochloride, 50 g
P0155	1, 10-Phenanthroline, 5 g

Consult the Flinn Scientific website for current prices.