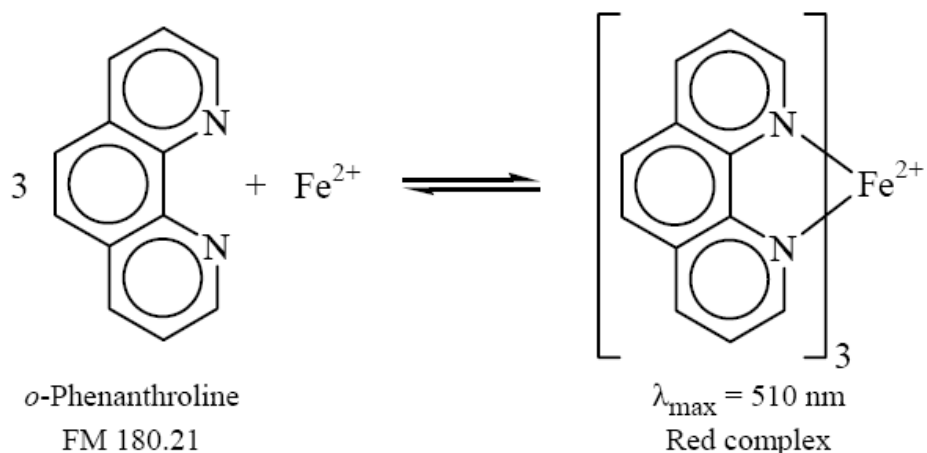


The Determination of Iron in a Natural Water

Discussion

The red-orange complex that forms between iron(II) and 1,10-phenanthroline (orthophenanthroline) is useful in determining iron in water supplies. The reagent is a weak base that reacts to form phenanthroline ion, phenH^+ , in acidic media. Complex formation with iron is described by the equation



The formation constant for this equilibrium is 2.5×10^6 at 25°C . Iron(II) is quantitatively complexed in the pH range between 3 and 9. A pH of about 3.5 is ordinarily recommended to prevent precipitation of iron salts, such as phosphates. An excess of a reducing reagent, such as hydroxylamine or hydroquinone, is needed to maintain iron in the +2 oxidation state. The complex, once formed, is very stable.

This determination can be performed with a spectrophotometer set at 508 nm or with a photometer equipped with a green filter.

PREPARATION OF SOLUTIONS

1. *Standard iron solution, 0.010 mg/mL.* Weigh (to the nearest 0.2 mg) 0.0702 g of reagent-grade $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \times 6\text{H}_2\text{O}$ into a 1-L volumetric flask. Dissolve in 50 mL of water that contains 1 to 2 mL of concentrated sulfuric acid; dilute to the mark and mix well.
2. *Hydroxylamine hydrochloride* (sufficient for 80 to 90 measurements). Dissolve 10 g of $\text{H}_2\text{NOH} \times \text{HCl}$ in about 100 mL of distilled water.

3. *Orthophenanthroline solution* (sufficient for 80 to 90 measurements). Dissolve 2.5 g of orthophenanthroline monohydrate in about 100 mL of water. Add water to make 1 L of solution. Each milliliter is sufficient for no more than about 0.2 mg of Fe. Prepare no more reagent than needed; it darkens on standing and must then be discarded.

4. *Sodium citrate, 2.5%*. Dissolve 25 g of $\text{Na}_3\text{Citrate} \times 2\text{H}_2\text{O}$ in 1 L of distilled water.

Preparation of the Calibration Curve

Transfer 25.00 mL of the standard iron solution to a 50-mL volumetric flask and 25 mL of distilled water to a second 50-mL volumetric flask. Add 1 mL of hydroxylamine, 5 mL of sodium citrate, and 4 mL of ortho-phenanthroline to each flask. Allow the mixtures to stand for 5 min; dilute to the mark and mix well. Clean a pair of matched cells for the instrument. Rinse each cell with at least three portions of the solution it is to contain. Determine the absorbance of the standard with respect to the blank.

Repeat this procedure with at least three other volumes of the standard iron solution; attempt to encompass an absorbance range between 0.1 and 1.0. Plot a calibration curve.

Determination of Iron

Transfer 10.00 mL of the unknown to a 50-mL volumetric flask; treat in exactly the same way as the standards, measuring the absorbance with respect to the blank.

Alter the volume of unknown taken to obtain absorbance measurements for replicate samples that are within the range of the calibration curve.

Report the concentration of iron in the unknown in parts per million (mg/L).