Brooklyn College
Department of Chemistry
Chemistry 41

Spectrophotometry

Determination of Iron

The Spectronic 21 spectrometer is used in this experiment to gain familiarity with analytical methods using ultraviolet-visible spectroscopy. The use of a complexing reagent to form an light-absorbing species is demonstrated. Iron(II) does not absorb visible light, but its complex with 1,10-phenanthroline forms colored solutions with an absorption maximum at 508 nm. The molar absorptivity of this complex is high, and this method is useful for determinations of iron at concentrations of a few ppm. The procedure that follows is based on the experiment in your text (Exp. 36M-2, p 859). The success of this procedure depends on using a reducing reagent (hydroxylamine) to convert iron(III) to iron(II) and using sodium acetate to adjust the pH to about 3.5 to prevent precipitation of iron salts.

Preparation of Solutions. (do not oven-dry the reagents used in this experiment)

- *Standard iron solution*. Weigh (to the nearest 0.2 mg) 0.14 g of reagent-grade $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ 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. This provides a solution that contains 0.02 mg/mL (or 20 ppm) of iron (twice the amount used in the text). Calculate the precise concentration of iron in your solution.
- *Unknown iron solution.* Weigh (to the nearest 0.2 mg) 0.10 g of the unknown 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.
- *Hydroxylamine hydrochloride*. Dissolve 1.0 g of $H_2NOH{\cdot}HCl$ in 10 mL of water (use a graduate cylinder).
- *Sodium acetate, 1.2 M*. Dissolve 17 g of $NaOAc{\cdot}3H_2O$ in 100 mL of water (graduate cylinder).
- *Orthophenanthroline solution*. A stock solution containing 1.0 g of orthophenanthroline monohydrate in 1.0 L of water will be provided. Obtain about 80 mL of this solution. (Do not use this solution if it has darkened.)

Procedure.

- Transfer 25.00 mL of the standard iron solution and 25.00 mL of the unknown iron solution to separate 100-mL volumetric flasks. Add 1 mL of hydroxylamine, 10 mL of sodium acetate, and 10 mL of orthophenanthroline solution to each flask. Allow the mixtures to stand for 5 min; dilute to the mark and mix well.
- Prepare three more standard solutions using 10.00, 5.00, and 2.00 mL (use a 10 mL delivery pipet) of the iron solution and the same volumes of the other reagents.
- Prepare a blank with no iron in it. (Start with 25 mL of water and add the other solutions as above.)

Determine the absorbance of each solution with respect to the blank. Rinse each cell with at least three portions of the solution it is to contain. Read Section 36M-1, p 858, in your text on the care and handling of cells. Specific directions for the instrument to be used will be provided in the lab.

Data Analysis and Report. Prepare a spreadsheet analysis of your data using the attached sample spreadsheet as a guide. This should include a graph of measured absorbance *vs.* concentration, a least-squares analysis of the data, and the percent iron in your unknown.