

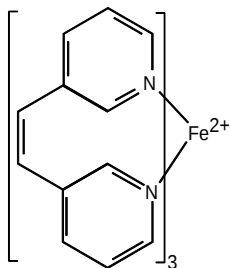
SPECTROPHOTOMETRIC DETERMINATION OF Fe IN DRINKING WATER

Background Reading: Harris, 7th ed., Chap. 18. Skoog, West, Holler and Crouch, 7th ed., Chap. 23A, pp. 594-601.

Introduction

Several contaminants in drinking water can be determined spectrophotometrically, including iron. Although iron is easily determined in contaminated water containing >1 ppm (1 mg/L), federal and state regulations limit the iron content of drinking water to <1 ppm. Thus, an intensely colored complex must be formed to detect the presence of these low levels of iron spectrophotometrically. As is the case whenever trace quantities of an analyte are being measured, **cleanliness of equipment, glassware, etc. is essential** to prevent positive determinate errors (false positives) due to laboratory contamination.

One widely used iron complex is iron(II)-o-phenanthroline, which is orange-red and easy to detect. Like most metal complexation reactions, the metal ion must compete with H_3O^+ ions, and thus the metal complex will not form in strongly acidic solutions. On the other hand, most metals form insoluble metal hydroxides in basic solutions. For these reasons the iron determination using o-phenanthroline is carried out in a **slightly** acidic solution.



Fe(II)-o-phenanthroline complex

In most water samples iron exists in its oxidized form, (Fe(III)), due to the presence of oxygen. Since it is the Fe(II) species that forms the complex with o-phenanthroline, a reduction must first be carried out. This can be accomplished by the addition of hydroxylamine. In the presence of an excess of hydroxylamine, the Fe(II)-o-phenanthroline complex is quite stable.

Your unknown is representative of a contaminated water sample. It has been spiked to produce an Fe concentration greater than 1 ppm Fe.

Procedure

Preparation of Stock and Standard Solutions. To prepare a 50 ppm stock solution of Fe, accurately weigh 175 mg of ferrous ammonium sulfate into a **clean** 500 mL volumetric flask. Dissolve in 100 mL of H_2O and then add 1 mL of concentrated H_2SO_4 . Dilute to the mark and mix thoroughly. Calculate the concentration of your stock solution, which should be close to 50 ppm.

To determine the number of drops of sodium citrate needed to bring the pH of standards to a value between 3 and 4, use a piper to transfer a 5 mL aliquot of the iron stock solution to a beaker and add a drop of bromophenol blue indicator. Add sodium citrate drop-by-drop until the indicator turns blue, noting the number of drops required. Discard this sample, and calculate the number of drops of citrate needed per milliliter of solution.

Obtain an unknown sample from the TA. Exchange a 100-mL volumetric flask for one that contains the unknown. Dilute to volume with deionized water. Record the unknown code from the label. Find the number of drops of citrate needed to bring the sample to pH 3-4: Transfer a 10 mL aliquot of the sample to a beaker and add a drop of bromophenol blue. Add either 25% sodium citrate (if the water is acidic) or 0.1 F H_2SO_4 (if the solution is basic) dropwise until the indicator reaches its transition color. Calculate the number of drops required per milliliter of sample and then discard this sample.

All standard and sample solutions should be prepared at the same time so that when the spectrophotometric measurement is made all solutions will have had equal time for color development, which is quite slow for this procedure. It is recommended that the actual absorbance measurement not be made until the following lab period.

To prepare colored Fe(II)-o-phenanthroline standards, transfer 1, 2, 5, and 10 mL aliquots of the

stock solution to 100 mL volumetric flasks using a Mohr pipet. Add 1 mL of hydroxylamine solution, 3 mL of o-phenanthroline solution, and the appropriate amount of citrate. Prepare a blank in the same way, omitting the stock iron solution and citrate. Dilute all solutions to the mark.

Transfer with a transfer pipet a 50 mL aliquot of the unknown water sample to a 100 mL volumetric flask. Add 1 mL of hydroxylamine solution, 3 mL of o-phenanthroline solution, and adjust the pH to 3-4 as described above. Dilute to the mark.

Absorbance Measurements. Turn on the ThermoSpectronic Genesis 20. The instrument must warm up for at least 20 minutes before measurements are made. Set the wavelength to 510 nm by using the nm up/down keys. Use the A/T/C key to put the instrument in the absorbance mode. The instrument cycles through modes, so press the button repeatedly until the absorbance mode is reached. Fill a cuvet with the blank solution and insert it into the spectrometer. Be sure to line up the mark on the cuvet with the arrow on the instrument so that the cuvet can be inserted in the same position for each measurement. Press the 0 ABS key. The display should read zero absorbance. Using the same cuvet, measure the absorbance of the standard and sample solutions. It is recommended that the measurements be made on the lowest concentration standard first and then on sequentially higher concentration solutions. Do this by removing the cuvet from the spectrometer, rinse it with the next solution to be measured, fill it with the solution to be measured, and place it back into the spectrometer.

Using Excel, prepare an analytical curve of absorbance vs. ppm iron. Have Excel add a trendline and report the equation of the line:

$$A = a \times C_{Fe} + b$$

where A is absorbance, C_{Fe} is the Fe concentration, a is slope and b is intercept of the linear regression line. Use the equation to determine ppm Fe of your drinking water sample. Don't forget the 50:100 dilution!

Report

Turn in your calibration graph, the equation of the calibration line, and the concentration of Fe (in ppm) in your sample.