CHEM241 Lab Handout
Expt. 8: THE DETERMINATION of IRON in VITAMIN TABLETS USING UV/VIS SPECTROSCOPY

Theory

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

$$Fe^{2+} + 3PhenH^+ \Leftrightarrow Fe(Phen)_3^{2+} + 3H^+$$

The formation constant for this equilibrium is 2.5×10^6 at 25^0C. 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 state. The complex, once formed, is very stable.

This determination can be performed with a spectrophotometer set at 510 nm or with a photometer equipped with a green filter.

Materials

Standard iron solution, 10.0 ppm
Hydroxylamine hydrochloride
1,10-phenanthroline solution
Sodium acetate, 1.2 M
Vitamin tablets

Procedure:

NOTE: You will be working in pairs. Each pair of students will analyze a brand of multivitamin tablets in *duplicate*.

A. Extraction of Fe from a multivitamin tablet

1) Place an iron-containing tablet in a 50-mL heat-resistant centrifuge tube. Add 25 mL of 6 M HCl. Place onto a ModBlock digester in the hood and boil gently for 15 min at a temperature of 95 ^{0}C.

2) Cool until it is just warm to the touch and gravity-filter into a 250-mL volumetric flask using flutted filter paper. Do this in the hood.

3) Perform a quantitative transfer by washing the centrifuge tube three times with a small amount of DI water and pouring the rinse water through the filter into the flask. Cool and dilute to the mark with DI water. Cap and mix well by inverting several times.

B. Spectroscopic Determination of Iron in Vitamin Tablets

(1) Transfer 1.00 mL of standard iron solution to a 25-mL volumetric flask. Add 0.25mL of hydroxylamine, 2.00 mL of sodium acetate, and 2.00 mL of 1,10-phenanthroline to the flask. Allow the mixture to stand for 5 min; dilute to the mark and mix well. Calculate the concentration of Fe in ppm (mg/L) in this diluted solution. Fill out Table 1.

Table 1. Blank, calibration standards, volumes of reagents used and new concentration of standards

Standard No.	Volume of 10.00 ppm Fe stock, mL	Volume of hydroxylamine. HCl, mL	Volume of sodium acetate, mL	Volume of 1,10-phenanthroline, mL	Total volume of solution, mL	Calculated concentration of iron, mg/L
Blank	0	0.25	2.00	2.00	25.00	
1	1.00	0.25	2.00	2.00	25.00	
2	2.00	0.25	2.00	2.00	25.00	
3	4.00	0.25	2.00	2.00	25.00	
4	5.00	0.25	2.00	2.00	25.00	

(2) Repeat the procedure above using 2.00 mL, 4.00 mL and 5.00 mL of standard iron solution on three separate 25mL volumetric flasks to prepare three other standard solutions. Do not forget to add the same amount of the other reagents as in step a. Calculate the ppm concentration of Fe in these dilutions and enter in Table 1.

(3) Prepare a blank by adding the reagents in step a to a 25-mL volumetric flasks, but omit the Fe solution. Dilute to the mark.

(4) Using a micropipet, transfer 0.750 mL of each vitamin solution to separate 25-mL volumetric flask; treat in exactly the same way as the standards by adding the same volumes of the other reagents. Complete Table 2 below except for the last column.

Table 2. Multivitamin solutions for iron analysis

Sample name	Volume of vitamin solution, mL	Volume of hydroxylamine .HCl, mL	Volume of sodium acetate, mL	Volume of 1,10-phenanthroline, mL	Total volume of solution, mL	Calculated mass of iron, mg
	0.75	0.25	2.00	2.00	25.00	
	0.75	0.25	2.00	2.00	25.00	

(5) Once the 5-minute reaction time is over, obtain 7 plastic *cuvettes* for the spectrometer. Transfer a portion of each of the prepared Fe and vitamin solutions.

(6) ***Spectroscopic Analysis:*** Determine the absorbance of *each standard solution* with respect to the blank at 510 nm following the operating instructions on the spectrometer for *quantitation*. Follow the steps for calibration and regression analysis.

(7) Measure the absorbance of each of the *vitamin solutions* with respect to the blank.

(8) After entering the appropriate dilution factor and using the line equation from the calibration curve, determine the concentration of iron in parts-per-million (mg/L) in the 250-mL solution after digestion. From this, calculate the amount of iron in milligrams per tablet.

(9) Check the multivitamin bottle label for iron content in milligrams and compare this value with your experimental value. Express the accuracy of your own results.