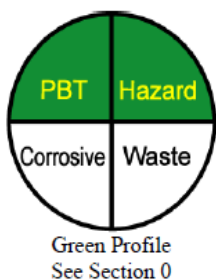
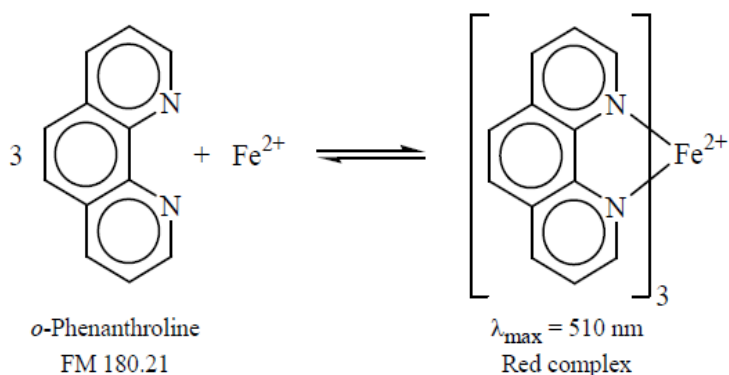
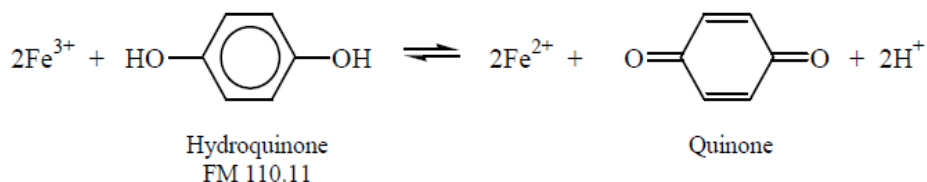


20. Spectrophotometric Determination of Iron in Vitamin Tablets³⁶



In this procedure, iron from a vitamin supplement tablet is dissolved in acid, reduced to Fe^{2+} with hydroquinone, and complexed with *o*-phenanthroline to form an intensely colored complex (Color Plate 15 in the textbook).



Reagents

Hydroquinone: (20 mL/student) Freshly prepared solution containing 10 g/L in distilled water.

Store in an amber bottle.

Trisodium citrate: (20 mL/student) 25 g/L $\text{Na}_2\text{citrate} \cdot 2\text{H}_2\text{O}$ (FM 294.10) in distilled water.

o-Phenanthroline: (25 mL/student) Dissolve 2.5 g in 100 mL of ethanol and add 900 mL of distilled water. Store in an amber bottle.

6 M HCl: (25 mL/student) Dilute 124 mL of 37 wt% HCl up to 250 mL with distilled water.

Standard Fe (40 $\mu\text{g Fe/mL}$): (35 mL/student) Dissolve 0.281 g of reagent-grade

$\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ (FM 392.14) in distilled water in a 1-L volumetric flask containing 1 mL of 98 wt% H_2SO_4 .

Procedure

1. Place one tablet of the iron-containing vitamin in a 125-mL flask or 100-mL beaker and boil gently (*in a fume hood*) with 25 mL of 6 M HCl for 15 min. Filter the solution directly into a

100-mL volumetric flask. Wash the beaker and filter several times with small portions of water to complete a quantitative transfer. Allow the solution to cool, dilute to the mark and mix well. Dilute 5.00 mL of this solution to 100.0 mL in a fresh volumetric flask. If the label indicates that the tablet contains <15 mg of Fe, use 10.00 mL instead of 5.00 mL.

2. Pipet 10.00 mL of standard Fe solution into a beaker and measure the pH (with pH paper or a glass electrode). Add sodium citrate solution 1 drop at a time until a pH of ~3.5 is reached. Count the drops needed. (It will require about 30 drops.)
3. Pipet a fresh 10.00-mL aliquot of Fe standard into a 100-mL volumetric flask and add the same number of drops of citrate solution as required in step 2. Add 2.00 mL of hydroquinone solution and 3.00 mL of *o*-phenanthroline solution, dilute to the mark with water, and mix well.
4. Prepare three more solutions from 5.00, 2.00, and 1.00 mL of Fe standard and prepare a blank containing no Fe. Use sodium citrate solution in proportion to the volume of Fe solution. (If 10 mL of Fe requires 30 drops of citrate solution, 5 mL of Fe requires 15 drops of citrate solution.)
5. Determine how many drops of citrate solution are needed to bring 10.00 mL of the iron tablet solution from step 1 to pH 3.5. This will require about 3.5 or 7 mL of citrate, depending on whether 5 or 10 mL of unknown was diluted in the second part of step 1.
6. Transfer 10.00 mL of solution from step 1 to a 100-mL volumetric flask. Add the required amount of citrate solution determined in step 5. Then add 2.00 mL of hydroquinone solution and 3.0 mL of *o*-phenanthroline solution. Dilute to the mark and mix well.
7. Allow the solutions to stand for at least 10 min. Then measure the absorbance of each solution at 510 nm in a 1-cm cell. (The color is stable, so all solutions may be prepared and all the absorbances measured at once.) Use distilled water in the reference cuvet and subtract the absorbance of the blank from the absorbance of the Fe standards.
8. Make a graph of absorbance versus micrograms of Fe in the standards. Find the slope and intercept (and standard deviations) by the method of least squares. Calculate the molarity of $\text{Fe}(\text{o-phenanthroline})_3^{2+}$ in each solution and find the average molar absorptivity (ϵ in Beer's law) from the four absorbances. (Remember that all the iron has been converted to the phenanthroline complex.)
9. Using the calibration curve, find the number of milligrams of Fe in the tablet. Use Equation 4-27 in the textbook to find the uncertainty in the number of milligrams of Fe.