Colorimetric Determination of Iron in Vitamin Tablets

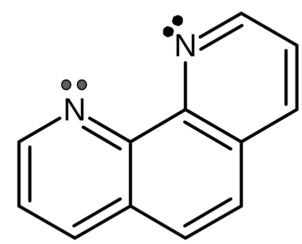
Cautions: 6 M hydrochloric acid is corrosive.

Purpose:

To colorimetrically determine the mass of iron present in commercial vitamin tablets using a prepared calibration curve.

Introduction:

Iron is considered an essential nutrient in the human body, having a role in the movement of oxygen through the blood as well as in some enzymatic actions. Iron can be found in various foods, including eggs, legumes, some whole grains, and red meat. In spite of these edible iron sources, many people suffer from dietary iron deficiencies. Therefore, iron supplements and fortified foods, such as cereal, are available to make up for these deficiencies. In this experiment, the quantity of iron in over-the-counter dietary supplements will be determined using colorimetry.



Although iron can exist in either a +2 or +3 oxidation state, the +2 ion is more biologically favored. In either state, iron ions form many coordination compounds; iron ions surrounded by ligands. *Ligands* are molecules that contain lone pairs of electrons that can coordinate to cations. When metal ion solutions are mixed with ligand solutions, a metal-ligand complex forms, indicated by a change in the color of the solution. In the present experiment 1,10-phenanthroline (phen, see structure right) will be used as the ligand; the nitrogen atoms provide the lone pairs of electrons that coordinate the iron ions. When an excess of phen is mixed with Fe^{2+}, a red solution results; the intensity of the color in solution will be a function of the concentration of Fe^{2+} present. The $Fe(phen)_3$ complex forms rapidly and is extremely stable, making it ideal for iron analysis. Hydroquinone is added to the prepared solutions to prevent oxidation of Fe^{2+} to Fe^{3+}.

Colorimetry is the measurement of the amount of color of a substance. Transition metal complexes often form highly colored solutions due to electrons moving between energy states in the metal center and states within the ligands. These electronic transitions have energies that correspond to wavelengths in the visible region (similar to the lines observed in the hydrogen atomic spectrum; see Ch 7 in Chang 8^{th} ed.). Therefore, when white light passes through a solution containing the metal complex, specific wavelengths will be absorbed which correspond to the energy of the electronic transitions.

Absorbance measurements are performed by comparing the amount of light that enters a chemical sample (I_0) to the amount of light that exits the sample (I). This ratio is better known as the transmittance:

$$T = \frac{I}{I_0} \qquad \textbf{(1)}$$

When a chemical sample absorbs radiation, I_0 is greater than I, making T a fraction. A more commonly used value is the absorbance:

$$A = -\log T \qquad \textbf{(2)}$$

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since this value can be directly related to the concentration of an absorbing species. This relationship is known as Beer's Law:

$$A = bce \qquad \textbf{(3)}$$

where *b* is the path length of the radiation through the sample (typically 1 cm), *c* is the concentration (in molarity) and e is the molar absorptivity, a proportionality constant that depends upon both the species and wavelength of light.

Based on Beer's law, a calibration curve can be prepared using standard solutions (those containing a known concentration of substance). A graph of the absorbance of these solutions vs. concentration should yield a straight line that passes through the origin. One requirement for performing quantitative analyses using calibration curves is that the absorbance of the unknown solution must fall within the absorbance values measured for the standard solution. This is further insurance that Beer's Law is truly being followed.

Above, Beer's Law is defined for the case where concentration is expressed in molarity. In practice, the linear relationship is valid for any concentration unit (e.g. ppm), however the proportionality constant can no longer be called the "molar" absorptivity (since concentration is no longer molar). In the present experiment, concentrations will be expressed in mg/L.

In this experiment the amount of iron in commercial vitamin tablets will be determined by colorimetry. A series of solutions of known concentration will be analyzed and a calibration curve will be prepared. Using the prepared curve and knowledge of dilutions, the concentration of iron in the original tablet will be determined.

Adapted from: D.C. Harris Quantitative Chemical Analysis, 5^{th} ed *Freeman Press, 1998 and R. C. Atkins,* J. Chem Ed. *52(1975)550.*

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Procedure:

Preparing the tablet solution

1. Your instructor will provide you with a vitamin tablet containing iron. Weigh your vitamin tablet, recording this mass and the identity of the tablet on your data sheet.

2. Crush the vitamin tablet using a mortar and pestle.

3. Transfer the crushed tablet to a 100 mL beaker, using 25 mL of 6 M HCl to rinse the residue on the mortar and pestle into the beaker. Boil this mixture *gently* on a hot plate in a hood for 15 minutes.

4. Gravity filter this solution directly into a 100 mL volumetric flask. Wash the beaker several times with small portions of deionized water to ensure complete transfer. Allow the solution to cool, then dilute to the mark with deionized water and mix well. Label this flask ***Tablet 1***.

5. Obtain a 50 mL volumetric flask and label it ***Tablet 2***. Use a volumetric pipette to transfer 2.50 mL of ***Tablet 1*** solution into this volumetric flask and dilute to the mark with deionized water.

6. Obtain another 50-mL volumetric flask, and label it ***Tablet 3***. use a volumetric pipette to transfer 5.00 mL of the solution labeled ***Tablet 2*** into this flask. Use graduated cylinders to add 1 mL citrate buffer, 1 mL hydroquinone solution, and 1.5 mL phenanthroline solution into this same flask. Dilute this solution to the mark with deionized water. ***Tablet 3*** is the sample solution to be analyzed.

Preparing standard solutions for calibration curve

7. Obtain a clean 50-mL beaker and label it "***Stock Iron Soln***". Pour about 20 mL of the stock iron solution provided by your instructor into this beaker. Make sure to record the concentration of this solution on your data sheet.

8. Obtain five 50-mL volumetric flasks. Label the flasks ***Blank, SS1, SS2, SS3, SS4***.

9. Use graduated cylinders to add 1 mL citrate buffer, 1 mL hydroquinone solution, and 1.5 mL phenanthroline solution to each of the flasks. The table below lists the proper volume of stock iron solution to deliver to each flask using a volumetric pipette. After adding all components, dilute to the mark with deionized water and mix well.

Flask	*Blank*	*SS1*	*SS2*	*SS3*	*SS4*
Volume of stock iron solution, mL	0.00	0.5	1.0	2.5	5.0

10. Allow all of the solutions to stand for at least 10 minutes.

11. Obtain 6 clean, dry cuvets for the Spec 20 from your instructor. Use a transfer pipette to fill each cuvet with a different solution (***Blank, SS1, SS2, SS3, SS4, Tablet 3***)

12. Set the wavelength measured by the Spec 20 to 508 nm. Measure the absorbance of each solution by placing the cuvets in the Spec 20 with the frosted sides facing you. Measure the blank first, then each of the samples in order. Finally, measure ***Tablet 3*** solution. Record all absorbance values in the appropriate spaces on your data sheet.

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Calculations:

1. Calculate the concentration of iron in each of the prepared standard solutions (***Blank, SS1, SS2, SS3, SS4***) using the dilution equation ($M_1V_1=M_2V_2$). Enter these values into the appropriate row of the Calibration Curve data table.
2. Prepare a calibration curve by graphing measured absorbance vs. concentration of iron using the information you entered in Calibration Curve data table.
3. Use the Calibration curve to estimate the concentration of iron in ***Tablet 3*** solution.
4. Use the dilution equation and the determined ***Tablet 3*** solution concentration to calculate the concentration of iron in the ***Tablet 1*** solution (HINT: you will have to use the dilution equation twice).
5. Finally, calculate the mass of iron present in the tablet using the concentration and volume of the ***Tablet 1*** solution.

<u>Calculations:</u>

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Data Sheets

Name: ______________________________________ **Lab Partner:** ____________________________

*Show work for all calculations on page 4***.**

Tablet Name: ____________________________

Mass of tablet (g)	
Mass of iron in tablet (from bottle) (mg)	
Concentration of stock iron solution (mg/L)	
Volume of “Tablet 1” solution (mL)	
Volume of “Tablet 2” solution (mL)	
Volume of “Tablet 3” solution (mL)	

Calibration Curve Data:

Flask	*Blank*	*SS1*	*SS2*	*SS3*	*SS4*
Volume of stock iron solution (mL)	0	0.5	1.0	2.5	5.0
[Fe^{2+}] in flask (mg/L)					
Measured absorbance					

Iron Tablet Data

Absorbance of “Tablet 3” solution	
[Fe^{2+}] “Tablet 3” solution (mg/L)	
[Fe^{2+}] “Tablet 1” solution (mg/L)	
Mass Fe in tablet (mg)	
Percent error	

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Post Lab Assignment

Name: ___________________________________

1. Did the calibration curve data obey Beer's Law? Explain why or why not.

2. List any additional sources of error (other than the quality of your calibration curve) in determining the mass of iron in the original iron tablet.

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Pre-Lab Assignment

Name: ____________________________________

A student performed this lab as described by the procedure and obtained the following results for the calibration curve and tablet solution:

Flask	***Blank***	***SS1***	***SS2***	***SS3***	***SS4***
$[Fe^{2+}]$ in flask (mg/L)	0	0.396	0.793	1.98	3.96
Measured absorbance	0.000	0.065	0.170	0.402	0.807

Absorbance of ***Tablet 3*** solution	0.182

a. Prepare a calibration curve of the data by graphing measured absorbance vs. $[Fe^{2+}]$. (Be sure to turn in this curve with your assignment.)

b. Use the calibration curve to determine the concentration of iron in the solution labeled ***Tablet 3***. Make sure to draw your interpolation lines.

c. Use the concentration obtained in part b and the procedure to determine concentration of iron in the ***Tablet 2*** solution.

d. Use the concentration obtained in part c and the procedure to determine concentration of iron in the ***Tablet 1*** solution.

e. Calculate the milligrams of iron in the tablet using the concentration of ***Tablet 1***.