Experiment 7B

ANALYSIS OF VITAMIN PILLS FOR IRON[1]

MATERIALS:

B & L Spectronic 20, 2 cuvette tubes, vitamin pill, 6 M HC1, filter paper, funnel, 5 mL graduated pipet, 25 mL graduated cylinder, 10 mL pipet, 2 100 mL beakers, watch glass, four 100 mL volumetric flasks, automatic burets, and the following solutions in burets: standard iron, o-phenanthroline, hydroquinone and sodium citrate.

DISCUSSION:

In this experiment you will analyze a substance containing only a very small amount of the material of interest, iron. A spectrophotometric technique will be used utilizing a reagent which greatly enhances the absorptivity of the substance to be analyzed. In the case of iron, a good reagent is o-phenanthroline. o-Phenanthroline forms a strong complex with Fe^{2+}, which absorbs light by a so-called charge transfer mechanism. This process is characterized by a large molar absorptivity, ϵ, in the order of thousands. Thus in the equation for Beer's Law,

$$A = \epsilon c \ell$$

(where A is the absorbance, ϵ is the molar absorptivity proportionality constant, c is the concentration in moles/liter and ℓ is the length of the path of light (in cm) through the solution), large absorbencies can be obtained with very low concentrations.

PROCEDURE: (work in pairs)

Note: Prior to use, rinse the 5 mL pipet with a small amount of the solution to be measured. In addition, rinse the cuvette with a few mL of the solution to be analyzed prior to measuring the percent transmission of the solution. Major errors may be introduced by failure to observe good technique, including proper rinsing of glassware.

Part A: Analysis of a Vitamin Pill

Before beginning on Part B, place a vitamin pill in a 100 mL beaker and add 25 mL of 6 M HC1. Cover the beaker with a watch glass and place it on the steam bath in the hood. While waiting for the vitamin pill/HC1 solution to boil, proceed to Part B.

Part B: Calibration Curve

Using the automatic buret, place 10.00 mL of the standard iron solution into a clean 100 mL volumetric flask. Add drop wise about 30 drops or about 1.5 mL of the sodium citrate solution. (This will give a solution with a pH of about 3.5.) Add 2.00 mL of hydroquinone solution[1] and 3.00 mL of o-phenanthroline (dispensed from burets). Dilute to the mark with distilled water and mix well. This solution now contains 8.00×10^{-5}M of Fe^{2+}. Prepare three other standard Fe^{2+} solutions by pipeting 5.00, 2.00, and 1.00 mL, respectively, of the standard iron solution into clean 100 mL volumetric flasks. <u>Add the appropriate amount of sodium citrate to each. Add 2 mL of hydroquinone and 3 mL of o-phenanthroline solution to each flask. Dilute to the mark with distilled water and mix well</u>. These four standard iron (Fe^{2+}) solutions should have the molar concentrations indicated in table 2.

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[1] The hydroquinone is a good reducing agent and will assure all iron is in the 2+ oxidation state.

Table 1 Summary of Components in Standard Solutions

Standard Solution	Standard Iron Solution	Sodium Citrate Solution	Hydroquinone Solution	o-Phenanthroline solution
1	10 mL	30 drops	2 mL	3 mL
2	5 mL	15 drops	2 mL	3 mL
3	2 mL	6 drops	2 mL	3 mL
4	1 mL	3 drops	2 mL	3 mL

After mixing, allow the standard solutions to stand 5 minutes and measure the percent transmittance of each solution at 508 nm against a blank of distilled water. Record the measured percent transmittance readings in Table 2 in the Data Section. Calculate the absorbance of each solution.

Plot a graph of absorbance versus concentration of Fe^{2+} in moles/L before you proceed to analyze the vitamin pill ("Part A Procedure continued"). There may be some disparity in the data, but in general, it will fall along a straight line.[2,3]
Submit this graph with your lab report[4].

Note: After your instructor has approved your calibration graph for Part B, empty the volumetric flasks and rinse thoroughly with tap water, followed by a few mL of distilled water. You will need to use these flasks for the remainder of the experiment.

Part A Procedure (continued):

One tablet of the brand vitamin to be analyzed was placed in a 100 mL beaker and heated to a slow boil with 25 mL of 6 M HC1 at the beginning of this experiment. Dilute this mixture slightly with water and filter it while hot through filter paper directly into a 100 mL volumetric flask. After washing the residue with hot water (available in large beakers on the steam baths), allow the filtrate to cool. Add distilled water up to the calibration mark. (Hereafter, this solution will be referred to as "Solution X".) Pipet a 5.00 mL aliquot into a clean 100 mL volumetric flask and diluted to volume with distilled water ("Solution Y"). Mix thoroughly.

Pipet a 10.00 mL aliquot of this new solution ("Solution Y") into a 100 mL volumetric flask. Add 3.5 mL sodium citrate, 2.00 mL hydroquinone and 3.00 mL o-phenanthroline and dilute the solution to the mark with distilled water ("Solution Z"). Again, mix well. After 5 minutes, measure the percent transmittance of the unknown solution ("Z") on the Spectronic 20. The absorbance of this unknown solution can then be calculated. The concentration of the Fe^{2+} in this final, twice diluted solution ("Z") can be determined by interpolation of the sample absorbance value on the standard calibration curve obtained in Part B.

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[2] For the most accurate work one should use a blank containing all of the above reagents (except iron) just in case they contain traces of iron. We do not do this in an effort to save time. If the graph in Part B is not a straight line, it is indicative of Fe being present in the various reagents.

[3] If the plot does not go through the origin, (0,0), it might be due to the use of different cuvettes to obtain the data or the presence of unknown amounts of Fe in the various reagents.

[4] Have your instructor check your graph before you proceed with the analysis of the vitamin pill. If there are major discrepancies in your data, your instructor may have you repeat some of your percent transmittance readings.

Name _____ Section _____

Partner _____ Date _____

DATA TREATMENT
Experiment 7B

Part B: Calibration Curve

Table 2 %T and Absorbance Readings of Standard Solutions

Standard Solution	Molarity Fe^{2+}	% Transmittance (measured)	Absorbance (calculated)
#1	8.00×10^{-5}		
#2	4.00×10^{-5}		
#3	1.60×10^{-5}		
#4	0.800×10^{-5}		

Part A: Analysis of a Vitamin Pill

Unknown solution of Vitamin Pill:

% Transmittance (measured): _____

Absorbance (calculated): _____

Molarity of Fe^{2+} in final,
twice diluted solution ("Z"): _____
(from graph of Part B)

Calculations:

1. Calculate the molarity of Fe^{2+} in the once diluted solution ("Y") of Part A.

2. Calculate the molarity of Fe^{2+} in the original 100 mL solution ("X") containing the vitamin pill.

3. Calculate the number of milligrams (mg) of iron in the vitamin pill.

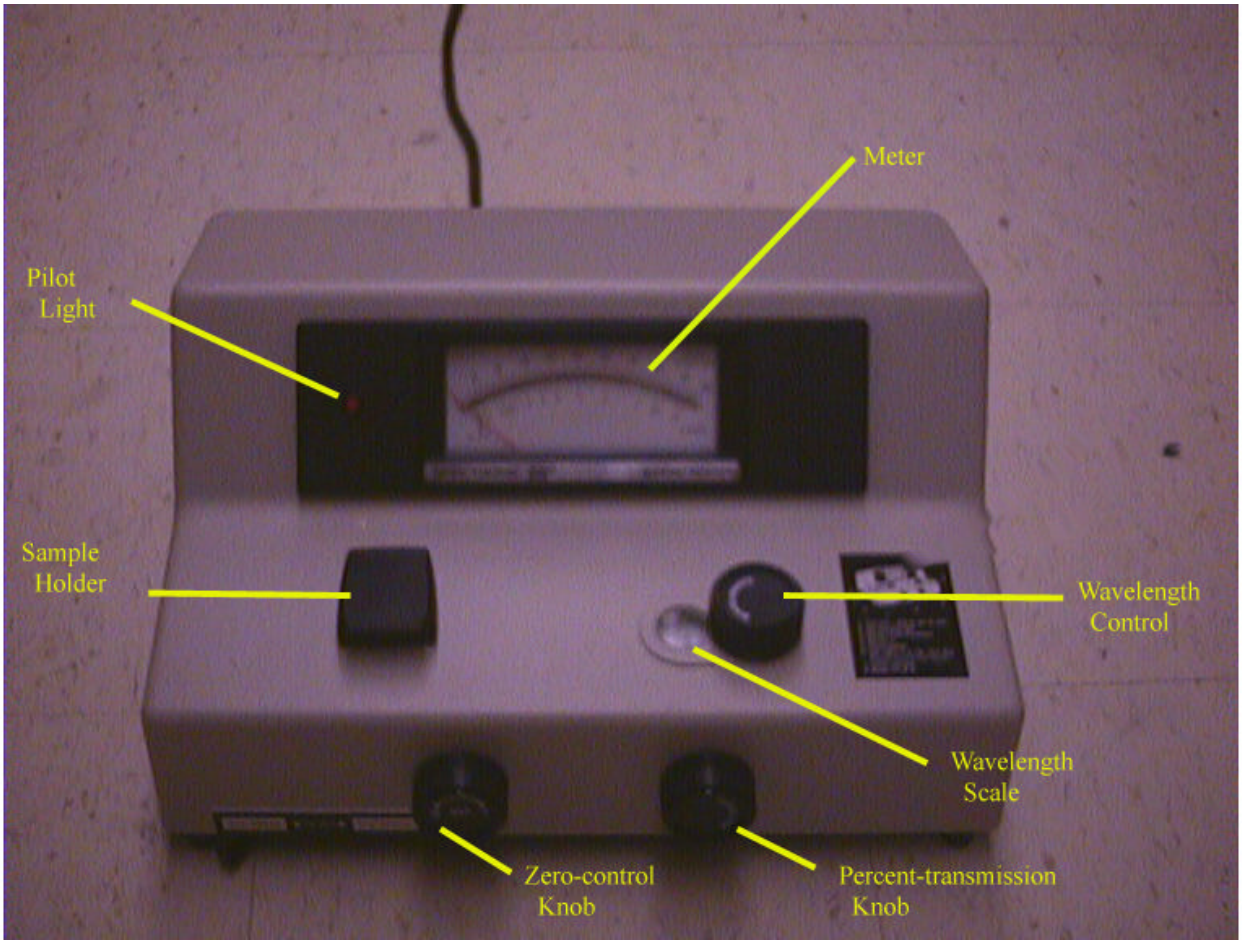
Notes:

1. See: Analytica Chimica Acta 9, 538 (1959) for more details.

Questions:

1. Calculate the value of the molar absorptivity, ϵ, for the Fe^{2+} o-phenanthroline complex, at 508 nm, using your absorbance vs concentration calibration graph. Assume a solution thickness of 1.0 cm. (HINT: See Appendix I.) The best slope of your graph can be obtained by using a computer spreadsheet, such as QUATTRO PRO, which handles data manipulation, graphing, and regression analysis (including linear regression).

2. Using the molar absorptivity determined in question 1, calculate the molarity of an iron complex solution whose percent transmittance at 508 nm equals 36.6. Assume a solution thickness of 0.92 cm.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