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### **CHEM 142 Experiment #4: Calibration Curves and an Application of Beer's Law**

Goals of this lab:

- Apply the use of a calibration curve to finding the concentration of an unknown
- Apply the mechanics of dimensional analysis to calculate the mass of iron in a sample based on concentration of an iron-containing solution
- Develop lab skills in operating digital pipettes, volumetric glassware, and spectrophotometers
- Use Excel to graphically represent and interpret experimental data
- Assess the accuracy of experimental data (compared to a known value) and identify sources of error

Your lab report will be grade on the following criteria using a poor/good/excellent rating system (see the Lab 4 Self-Assessment for more details):

- Calculations are complete and correct, with proper use of significant figures and units
- Data and results are careful and accurate
- Lab report is clear, legible, and neat
- Error analysis is well-supported and valid
- All graphs and tables are clearly and accurately labeled; entire report is typed
- Application of skills to new situations is accurate and complete

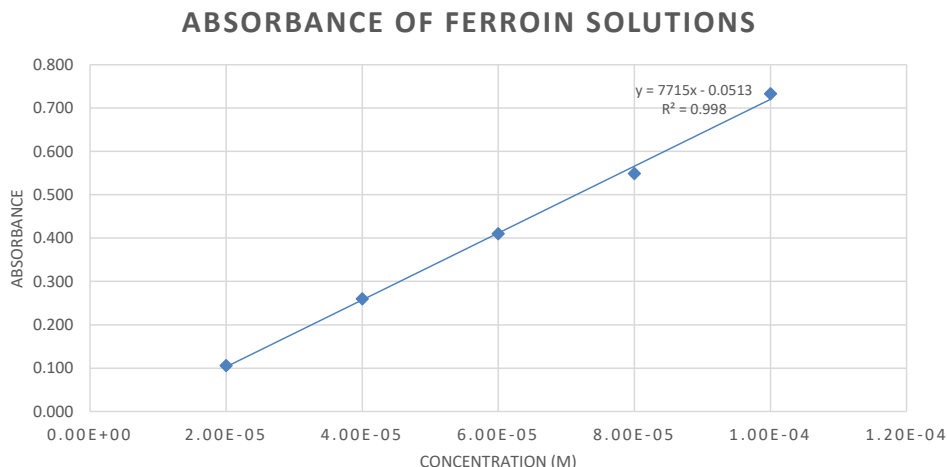
**By signing below, you certify that you have not falsified data, that you have not plagiarized any part of this lab report, and that all calculations and responses other than the reporting of raw data are your own independent work. Failure to sign this declaration will result in 5 points being deducted from your lab score.**

Signature: Naeco Pasternak \_\_\_\_\_

*This lab is worth 60 points: 10 points for notebook pages, 50 points for the lab report*

**DATA, GRAPHS AND CALCULATIONS****Creating the calibration curve:** $\lambda_{\text{max}}$  for absorbance measurements:  nm (from Part III. B.)**Note:****All sections of this report must be typed**Ferroin Standards:  
(from Part III. C.)

Concentration (M)	Absorbance
2.00E-05	0.106
4.00E-05	0.260
6.00E-05	0.410
8.00E-05	0.549
1.00E-04	0.733

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Slope of Absorbance versus concentration graph

  $\text{M}^{-1}$ 

(enter #s here so the data

y-intercept of Absorbance versus concentration graph

will correctly autofill on pg 3)

Detailed calibration equation:

(review the introductory information in the lab manual for an explanation of what is meant by a "detailed" calibration equation)

**Determining the Amount of Iron in an Iron Tablet**

- Average mass of a tablet  mg
- Mass of crushed tablet used in analysis  mg (enter a # here so the data will correctly autofill on pg 3)
- Final volume after filtered crushed tablet solution is diluted in volumetric flask (lab manual Part II, Step 5)  mL
- Volume of diluted crushed tablet solution transferred to the new volumetric flask (lab manual Part II, Step 6)  mL
- Final volume of ferroin complex solution (lab manual Part II, Step 9)  mL
- Absorbance of the ferroin complex solution (lab manual Part III, Step C.7)  (enter a # here so the data will correctly autofill on pg 3)

Student- specific data from pg 2 used in calculations autofill here on this page:

Calib. Curve slope:	7715	y-int of Calib. Curve:	-0.051
Absorbance of digested sample:	0.547	Mass crushed tablet:	59.0

- 7) Using the calibration equation and the absorbance you measured for the prepared sample, calculate the ferroin concentration. Show your work and don't forget to include units.
- Absorbance = 7715 ferroin (M) + -0.0513  
 $0.547 = 7715 \text{ ferroin (M)} + -0.0513$   
 $0.5983 = 7715 \text{ ferroin (M)}$   
 $0.5983 / 7715 = \text{ferroin (M)} = 0.00007755022 = 7.76 \times 10^{-5} \text{ ferroin (M)}$
- 8) Based on the procedural steps and the ferroin concentration you just calculated, calculate the moles of ferroin in the final ferroin complex solution prepared in Part II, Step 9. Show your work, including units.
- moles = Molar concentration/volume  
 $\text{moles} = 7.76 \times 10^{-5} \text{ ferroin (M)} / 0.1 \text{ L}$   
 $\text{moles} = 7.76 \times 10^{-5} \text{ moles of ferroin}$
- 9) Based on the moles of ferroin in the final ferroin complex solution, calculate the moles of iron in the crushed tablet solution prepared in Part II, Steps 2-5. Show your work, including units.
- Final solution uses just 5 mL of the initial 100mL of solution  
 $100 \text{ mL} / 5 \text{ mL} = 20 \text{ times as many moles in the crushed tablet solution from steps 2-5.}$   
 $(7.76 \times 10^{-5} \text{ moles of ferroin}) \times 20 = 0.001552 \text{ moles} = 1.55 \times 10^{-3} \text{ moles}$
- 10) Using the "moles of iron in the crushed tablet solution" you just calculated, calculate the mass (in mg) of iron in the crushed sample that you weighed out. Show your work, including units.
- $1.55 \times 10^{-3} \text{ moles Fe} \times (55.845 \text{ g Fe} / 1 \text{ mol Fe}) = 0.08655975 \text{ g Fe} = 86.6 \text{ mg Fe}$
- 11) From the mass of iron in the crushed tablet sample you weighed out, calculate the mass (in mg) of iron in a whole tablet. Show your work, including units.
- Average mass of tablet = 436 mg, 59 mg of crushed iron tablet used in lab  
 $436 \text{ mg} / 59 \text{ mg} = 7.38983$   
 $7.38983 \times 86.6 \text{ mg Fe} = 639.661881365 \text{ mg Fe} = 639 \text{ mg Fe}$
- 12) mg of iron per tablet (as listed on the bottle) 50 mg

## Results and Discussion

1. Compare your mass of iron per tablet with the amount listed on the bottle label. Calculate the % error and discuss YOUR major sources of error. How did this affect your results?

Mass on bottle = 500 mg

% error =  $|( \text{experimental} - \text{actual} ) / \text{actual} | \times 100$

% error =  $|( 639 \text{ mg} - 500 \text{ mg} ) / 500 \text{ mg} | \times 100$

% error =  $|-0.278| \times 100$

% error = 27.8 %

My major source of error would have been in the dilution process. This process leaves a lot of room for variation, depending on how much from another solution was used in the transformation. Another major source of error could

depending on how much iron or other solutes were lost in the transfer process. Another major source of error could be within my lab notebook. I could've misread any of the data that I recorded within the lab, which I highly suspect,

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2. If you did not wait for the complete formation of the ferroin complex in Part II, step 10, how would your Abs data be different? Explain how would this affect your determination of the mass of iron in the tablet?

*The absorbance would've been lower on the iron solution sample. The 1,10 phenanthroline would not have reacted fully with the iron solution. This means that the first step in the process of finding the mass, substituting its absorbance for the absorbance in the calibration equation, would be incorrect, meaning that all following calculations for finding the mass would also be incorrect, as it would be based on the faulty result of the first step.*

3. You use atomic emission spectroscopy, another spectroscopic technique, to measure the  $\text{Li}^+$  concentration in 5 standard solutions of varying concentrations of  $\text{LiCl}$ . The intensities for the standard solutions are plotted versus the concentrations and the resulting calibration equation is:  $\text{Intensity} = 82,985 \text{ M}^{-1} * [\text{Li}^+] + 2.15$

If the intensity of your unknown sample is 132, what is the concentration of  $\text{Li}^+$  in the analyzed sample?

$$\text{Intensity} = 82,985 \text{ M}^{-1} * [\text{Li}^+] + 2.15$$

$$132 = 82,985 \text{ M}^{-1} * [\text{Li}^+] + 2.15$$

$$\text{M}^{-1} = 1.565$$

*The concentration would be  $1.565 \text{ M}^{-1}$ .*

If 15 mL of the original unknown sample was diluted to 375 mL prior to analysis, what is the concentration of  $\text{Li}^+$  in the original solution?

$$15 \text{ mL} * x = 375 \text{ mL} * 1.565 \text{ M}^{-1}$$

$$0.015 \text{ L} * x = 0.375 \text{ L} * 1.565 \text{ M}^{-1}$$

$$x = (0.375 \text{ L} * 1.565 \text{ M}^{-1}) / 0.015 \text{ L} = 39.125 \text{ M}^{-1}$$

*$39.125 \text{ M}^{-1}$  is the concentration of the original solution.*

### Laboratory Waste Evaluation

Laboratory waste is considered *anything* generated during an experiment that is disposed of down the sewer drain, thrown in the garbage, collected in a container for disposal by the UW Environmental Health & Safety department, or released into the environment. Based on the written lab procedure *and* your actions during the lab, list the identity and approximate amount (mass or volume) of waste that you generated while performing this experiment.

10 ml ferroin solution \* 5 (total solutions) \* 2 (rinse) = 100 ml of ferroin solutions

59 mg iron tablet + 4ml 0.10 M HCL + 10 mL DI water + 10 mL DI water + 70-80 mL DI water, take 5ml of it and put it in 100mL volumetric flask + 2mL 0.23 M hydroxylamine hydrochloride + 1 mL 1.0 M sodium acetate + 5mL  $3.6 * 10^{-3} \text{ M}$  + 87 mL DI water, total of this part = 199 mL Total waste: 100 mL ferroin solutions + 199 mL liquid waste = 299 mL liquid waste