

Lab 4. Determination of the Equilibrium Constant for the Iron (III) thiocyanate Reaction

Prelab Assignment

Before coming to lab:

- After reading "Lab Notebook Policy and Format for Lab Reports" handout, complete in, your lab notebook the following sections of the report for this lab exercise: Title, Introduction, Materials/Methods and Data Tables. An outline or flow chart of the procedure is appropriate for the Materials/Methods section. Ensure that the table of contents of your lab notebook is current.
- Read the lab thoroughly and answer the pre-lab questions that appear at the end of this lab exercise. Background information for this lab can be found in [Chapter 17](#) in your textbook (*Silberberg 6th ed*).

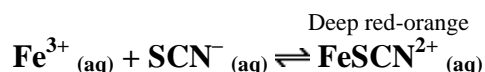
Purpose

The equilibrium for the reaction of iron (III), Fe^{3+} , with thiocyanate, SCN^- , to yield the colored product, iron (III) thiocyanate, FeSCN^{2+} , will be studied and its equilibrium constant determined spectroscopically using a *Vernier Ocean Optics Spectrometer*. A Beer's law plot will be made for iron (III) thiocyanate. Using a set of different starting concentrations and measuring the concentration of iron (III) thiocyanate spectroscopically, the equilibrium constant for the reaction will be determined.

Introduction

When a chemical reaction reaches **equilibrium** the concentrations of reactants and products no longer change with time. Thus, if one is able to measure these concentrations, the value of the equilibrium constant can be easily calculated.

In this laboratory you will find the equilibrium constant for the reaction:



Since the product, FeSCN^{2+} , has a deep red-orange color, its concentration can be determined using spectrophotometric techniques we have used in the past.

Once the equilibrium concentration of FeSCN^{2+} has been determined, the equilibrium concentrations of the reactants (Fe^{3+} and SCN^-) can be calculated. Because the stoichiometry is 1 mol Fe^{3+} : 1 mol SCN^- : 1 mol FeSCN^{2+} , the moles of each reactant used up in the reaction is equal to the moles of product formed. And since you know the initial concentrations of the reactants, it is a simple subtraction to find the moles of reactant remaining at equilibrium. Finally, once you know the concentration of each substance in the reaction, you can calculate the equilibrium constant, where

$$K_{\text{eq}} = \frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^{-}]}$$

In this experiment you will carry out the reaction several times, each time combining different amounts of the reactants, Fe^{3+} and SCN^- . The experimental equilibrium constant will then be found by averaging the results from all the trials.

As was the case in previous experiments where you conducted spectrophotometric measurements, you will first need to prepare a calibration curve using known solutions of

FeSCN^{2+} . In order to prepare the calibration curve you will need to prepare several solutions with known FeSCN^{2+} concentrations. However, it may not be so obvious how this can be accomplished when any mixture of the reactant ions results in an equilibrium mixture of all three ions. Fortunately, there is a way to combine Fe^{3+} and SCN^- such that the final concentration of FeSCN^{2+} can be known with certainty. The answer relies on ***Le Châtelier's principle***, which tells us that the equilibrium can be shifted far to the right if a very large excess of one reactant is used. Thus, to prepare the standard solutions, you will combine a limiting amount of Fe^{3+} with a large excess of SCN^- . This excess of SCN^- will force the equilibrium to the right, effectively converting all of the Fe^{3+} to FeSCN^{2+} .

Once your calibration curve has been prepared you will be able to prepare a series of equilibrium mixtures and determine the equilibrium constants for each trial, as outlined above. *Prior to carrying out the trials consider how the equilibrium constant will vary with each. What is your hypothesis? (See prelab questions!)*

Procedure (In this experiment you are to work in teams at each lab bench.)

- Preparation of 100.0 mL $1.25 \times 10^{-4} \text{ M Fe(NO}_3)_3$ diluted in 0.1 M HNO_3**
 - Obtain about **175 mL** of **0.1 M HNO_3** in a small clean and dry beaker. This should be enough for both Parts I and II below. Rinse a buret (the tip too!) two times with small portions (~5 mL), and then fill the buret. You will use the buret to measure the appropriate amounts later in Part I and II according to the tables below.
 - A stock solution of **0.00250 M $\text{Fe(NO}_3)_3$** in 0.1 M HNO_3 is available on the lab cart. Obtain about **50 mL** in a small clean and dry beaker—this is enough for both parts I and II.) Use the appropriate size *pipet* and a volumetric flask to prepare **100.0 mL** of a **$1.25 \times 10^{-4} \text{ M Fe(NO}_3)_3$** solution, using 0.1 M HNO_3 for the dilution. **See prelab question!** Mix well and label. *This diluted solution will only be used in Part I.*
- Obtain about **45 mL** of **1.00 M KSCN** in a small labeled beaker—*this more concentrated solution will only be used in Part I.* A more dilute solution of KSCN (0.0025 M) is also available on the lab cart. Be sure to use the correct solution!

Part I — Preparation of the Calibration Curve

- Set up five clean and dry test tubes in a rack and number them A1–A5. Use *pipets* to measure the solutions of Fe^{3+} , SCN^- , and use the *buret* to measure HNO_3 in the proportions listed below. **Be sure to use the correct concentration solutions!** Carefully use the vortex machine to insure that the solutions are well mixed.

Test Tube No.	$[\text{FeSCN}^{2+}]$ mol/L	$1.25 \times 10^{-4} \text{ M Fe(NO}_3)_3$ (mL)	1.00 M KSCN (mL)	0.1 M HNO_3 (mL)	Absorbance
1		1.00	5.00	4.00	
2		2.00	5.00	3.00	
3		3.00	5.00	2.00	
4		4.00	5.00	1.00	
5		5.00	5.00	0.00	

- Use the spectrophotometer to find the λ_{max} wavelength of solution 5 (it should be close to 460 nm) and measure the absorbance of each solution at this wavelength. Make sure you record both the wavelength and each absorbance in your notebook.
- Calculate and record in your lab notebook the $[\text{FeSCN}^{2+}]$ in mol/L in each test tube in Part I before coming to lab. *Because a large excess of SCN^- is used, it is reasonable to assume that all of the Fe^{3+} is converted to FeSCN^{2+} . So which reactant is limiting? Be sure to take into account the dilution that occurs when the solutions are mixed. See prelab questions!*
- Plot absorbance vs. $[\text{FeSCN}^{2+}]$ using *LoggerPro* or *Excel*, and confirm that the points fall along a straight line. You will need the equation for the linear fit and R^2 and a copy of the calibration curve *See Analysis point 1*.

Part II — Determination of the Equilibrium Constant

- Note: You will no longer need the **$1.25 \times 10^{-4} \text{ M Fe(NO}_3)_3$** or the **$1.00 \text{ M KSCN}$** solutions used in Part I. Set these aside so you don't use them by mistake.
- Set up 10 clean and dry medium size test tubes in a rack and label them B1–B10.
- Use 1-mL or 2-ml pipets to measure out the stock **$0.00250 \text{ M Fe(NO}_3)_3$** solution into each test tube according to the amounts listed in the table below. **Be sure to use the correct solution**—you will no longer need the diluted $\text{Fe(NO}_3)_3$ solution you made in Part I!
- Obtain **45 mL** of **0.00250 M KSCN** in a clean and dry beaker. Use small portions (<5 mL) of **0.00250 M KSCN** to rinse a buret (including the tip) two times, and then fill the buret with the remainder—make sure you rinse and fill the tip. The buret does not need to be filled to the zero mark – you will only need 20 mL to do all the trials. **Again, make sure you are using the correct solution.** Use the buret to measure the amounts of KSCN listed below.
- Refill the buret containing **0.1 M HNO_3** and use this buret to measure the amounts listed in the table below—the buret does not need to be filled to the zero mark since you will need only 35 mL to do all the trials.
- Add the solutions of Fe^{+3} , SCN^- , and HNO_3 in the proportions listed below. Carefully use the Vortex machine to insure that the solutions are well mixed.
- Measure and record the absorbance of each solution at your λ_{max} . Ideally you should run these solutions immediately after the standards in part I.

Test Tube No.	Absorbance	$0.00250 \text{ M Fe(NO}_3)_3$ (mL)	0.00250 M KSCN (mL)	0.1 M HNO_3(mL)
1		1.00	1.00	5.00
2		1.00	1.50	4.50
3		1.00	2.00	4.00
4		1.00	2.50	3.50
5		1.00	3.00	3.00
6		2.00	1.00	4.00
7		2.00	1.50	3.50
8		2.00	2.00	3.00
9		2.00	2.50	2.50
10		2.00	3.00	2.00

Analysis

1. Use *LoggerPro* or *Excel* to prepare a Beer's Law plot of absorbance vs. concentration for the 5 standard solutions in Part I. Plot the best-fit line (trendline) and give the equation and R^2 .
2. You should set up an *Excel* spreadsheet to do all of the following calculations for you. The following table summarizes the calculations. For test tube #1 in Part II, show in your notebook a complete sample calculation by hand including an ICE table.
3. First, compute the initial $[\text{Fe}^{3+}]$ and $[\text{SCN}^-]$ in each test tube for Part II. Be sure to take into account the dilution that occurs when the solutions are mixed.
4. Next, calculate the equilibrium $[\text{FeSCN}^{2+}]$ for Part II from the measured absorbance and the equation of the Beer's Law plot.
5. Based on the amount of FeSCN^{2+} formed (*i.e.*, the equilibrium concentration) determine:
 - a. the amount of both Fe^{3+} and SCN^- that reacted, and
 - b. the equilibrium concentrations of both Fe^{3+} and SCN^-

Again, use an Excel spreadsheet to do these calculations for you. The following table summarizes the calculations. Show in your notebook an ICE table with sample calculations by hand for Test Tube #1 in Part II.

Concentrations	$[\text{Fe}^{3+}]$ & $[\text{SCN}^-]$	$[\text{FeSCN}^{2+}]$
Initial	Calculated using initial concentrations and accounting for dilution	= 0
Change	Each amount of Fe^{3+} & SCN^- reacted = amount of FeSCN^{2+} formed	= Equilibrium value below
Equilibrium	= Initial – Change	Determined spectrophotometrically from Beer's law plot

6. Once the equilibrium concentrations of all species have been determined, calculate the equilibrium constant for each of the 10 trials. Then compute the average and standard deviation among the ten test tubes—all of these calculations should be done using Excel, but include in your notebook a sample calculation for test tube # 1 in part II.

As always, include a thoughtful and detailed discussion of your results in the Analysis section of your lab report. Examples of topics for discussion would be how well Beer's Law is obeyed and the use of Le Châtelier's principle, the precision (R^2 in Part I and ST DEV in Part II) of your results, the meaning of your average K_{eq} , and as always the major sources of error. If your instructor provides you with the theoretical value for K_{eq} , then calculate the % error and discuss the most likely sources of error that contributed to your values being too high or too low. Don't forget to end your report with a **conclusion section**!

Acknowledgement: This lab is adapted from similar labs produced by my colleagues at GRCC.

Lab 4 Report Checklist...

Introduction

- Includes goals of the experiment?
- Discusses Background info, including the chemical and algebraic equations and the use of L.C.P?

Materials/Methods

- Procedure neatly recorded and easy to follow for Parts 1 & 2?
- Enough detail so that a competent student could duplicate the experiment?

Results

- Data is neatly recorded in a ruled and easy to read table or tables?
- Tables numbered consecutively and have captions?
- Correct use of sig figs and units?
- **Part 1.** Absorbance data for calibration Curve
- **Part 2.** Absorbance data for tubes 1-10

Analysis of Results

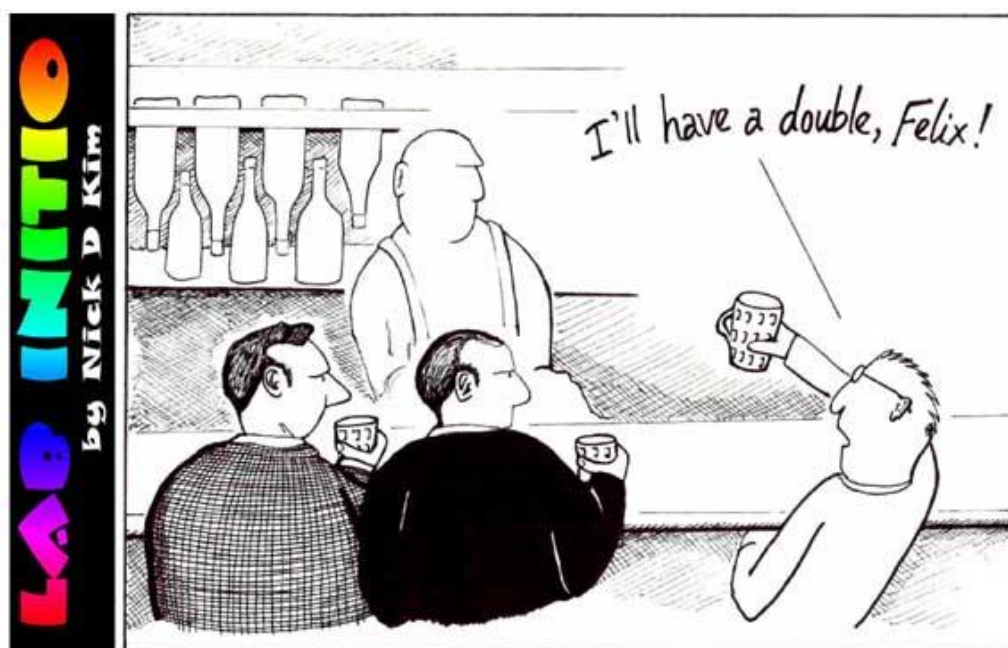
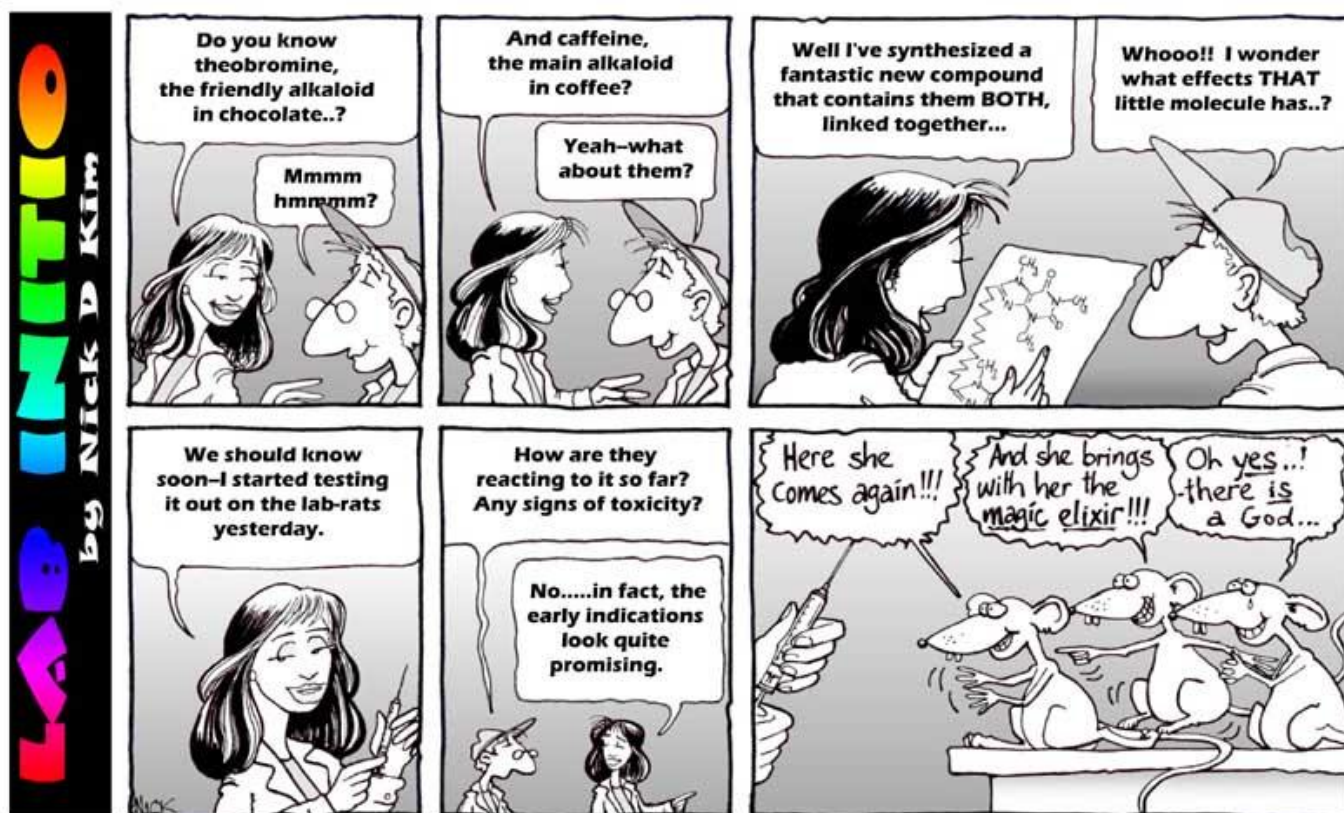
- **Excel or Logger Pro Plot** of Calibration Curve: **A vs. $[\text{FeSCN}^{+2}]$**
 - Graph has an informative caption?
 - Axes labeled and correct use of sig figs?
 - Equation of trendline and R^2 on graph?
- Sample calculations for test tube #1 in part 2 of ...
 - initial $[\text{Fe}^{+3}]$ and $[\text{SCN}^-]$
 - $[\text{FeSCN}^{+2}]_{\text{equil}}$ using equation of line from calibration plot
 - equilibrium $[\text{Fe}^{+3}]$ and $[\text{SCN}^-]$
 - K_{eq}
 - Correct use of units and sig figs?
- Data and all calculations summarized in an Excel table?
- % Error and error analysis?

Conclusion

- Uses “*bullets*” to state concisely the major conclusions
- Value for K_{eq}
 - % Error
 - Summarizes sources of error or uncertainty?

Just for fun...

Chemistry in Action...



Cambridge, 1953. Shortly before discovering the structure of DNA, Watson and Crick, depressed by their lack of progress, visit the local pub.

Lab 4. Determination of an Equilibrium Constant Name _____
Prelab Questions Team Number _____

Instructions: Complete the following five questions and hand in at the start of your lab period or when instructed by your instructor. Show your work with units and correct significant figures for all questions that involve a calculation.

It will be helpful to read the entire experiment first before attempting the following questions.

1. Why will the absorbance measurements be done at a wavelength of about 460 nm? What color in 460 nm? What color of light is absorbed?
2. What is your hypothesis for how the equilibrium constant will vary with the trials in Part II?
3. State in detail how you will prepare a 100.0 mL of a 0.000125 M $\text{Fe}(\text{NO}_3)_3$ solution from a stock solution of 0.00250 M $\text{Fe}(\text{NO}_3)_3$, using a volumetric flask and diluted with 0.1 M HNO_3 as described in step 1b on page 2.
4. Calculate the $[\text{FeSCN}^{2+}]$ in mol/L in each test tube in Part 1 and enter your results in the table below and in the table you prepared in your notebook. Show a sample calculation for test tube #1 only.

Test Tube No.	$[\text{FeSCN}^{2+}]$ mol/L	1.25×10^{-4} M $\text{Fe}(\text{NO}_3)_3$ (mL)	1.00 M KSCN (mL)	0.1 M HNO_3 (mL)
1		1.00	5.00	4.00
2		2.00	5.00	3.00
3		3.00	5.00	2.00
4		4.00	5.00	1.00
5		5.00	5.00	0.00

Sample calculation for test tube #1:

☺ ☺ ☺ Please turn the page for more pleasure! ☺ ☺ ☺

5. Ions A^{3+} and B^{1-} react to form the complex ion, AB_2^{1+} : $A^{3+}_{(aq)} + 2 B^{1-}_{(aq)} \rightleftharpoons AB_2^{1+}_{(aq)}$
 If 10.0 mL of **0.300 M** A^{3+} added to 10.0 mL of **0.600 M** B^{1-} , at equilibrium **1.88×10^{-3} mole** of AB_2^{1+} is present. Determine the equilibrium constant for this reaction. Complete the ICE table and show your work below. Circle your answer.

	$[A^{3+}]$	$[B^{1-}]$	$[AB_2^{1+}]$
I			
C			
E			