DETERMINATION OF AN EQUILIBRIUM CONSTANT

INTRODUCTION

In this experiment you will be determining the equilibrium constant, K_c, for the chemical reaction

$$Fe^{3+}(aq) + SCN^-(aq) \leftrightarrow FeNCS^{2+}(aq)$$
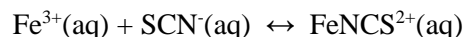
by measuring the concentration of $FeNCS^{2+}$ at equilibrium using a spectrometer. The equilibrium constant expression for this reaction is

$$K_c = \frac{[FeNCS^{2+}]}{[Fe^{3+}][SCN^-]}$$

PROCEDURE

1. Double click the "Logger Pro" icon and allow the screen to open.

2. The Spectrometer needs to be powered for about 5 minutes before using so do this step before preparing your solutions. Do not use the Go!Link with the spectrometer. Plug the Spectrometer via provided USB cable to the computer USB port.

3. Prepare standard calibration solutions according to Table 1.
   ***Note: In this part the solutions are prepared so that the initial Fe^{3+} concentration is much greater than the initial SCN^- concentration. Under these conditions the equilibrium position lies far to the right and it can be assumed that essentially all of the SCN^- has reacted to form $FeNCS^{2+}$.

   a. Use a pipet to deliver the **0.0020 M NaSCN** into clean, dry labeled large test tubes (TT).

   b. The standard Fe solution (**Fe(NO₃)₃ ; 0.20 M**) is in a preset (2.5 mL) pump dispenser.

   c. Using a buret, dispense the volume of 0.25 M nitric acid needed to bring the total volume to 10.0 mL.

Table 1.

Solution	0.0020 M NaSCN (in 0.25 M HNO₃)	0.20 M Fe(NO₃)₃ (in 0.25 M HNO₃)	0.25 M HNO₃	[FeNCS²⁺]
1 (blank)	0 mL	2.5 mL	dilute to 10.0 mL	0
2	0.20 mL	2.5 mL	dilute to 10.0 mL	
3	0.50 mL	2.5 mL	dilute to 10.0 mL	
4	1.0 mL	2.5 mL	dilute to 10.0 mL	
5	1.5 mL	2.5 mL	dilute to 10.0 mL	3.0 x 10⁻⁴ M

4. Prepare solutions according to Table 2.

   a. Determine the number of moles of Fe^{3+}, SCN^-, and $FeNCS^{2+}$ that will be **initially** present for each solution in Table 2 and enter these in the appropriate places in the five tables (Table 2: A-E located in the data analysis section of this experiment).

   b. Use a pipet to deliver the **0.0020 M NaSCN** into clean, dry labeled large test tubes.

   c. The **0.0020 M Fe(NO₃)₃** solution is in a preset (5.0 mL) pump dispenser.

   d. Deliver the 0.25 M nitric acid from a buret.

Table 2.

Solution	0.0020 M NaSCN (in 0.25 M HNO₃)	0.0020 M Fe(NO₃)₃ (in 0.25 M HNO₃)	0.25 M HNO₃
A	1 mL	5 mL	4 mL
B	2 mL	5 mL	3 mL
C	3 mL	5 mL	2 mL
D	4 mL	5 mL	1 mL
E	5 mL	5 mL	0 mL

5. Calibrate the Spectrometer ; Do not unplug the spectrometer during this experiment or you will have to start over.

   a. Return to the Logger Pro screen on the computer. Click: Experiment ; Calibrate ; Spectrometer:1.

   b. Allow the lamp to warm up for 90 seconds as displayed on the computer screen.

   c. Only touch the ridged faces of the cuvette, never touch the clear faces. Rinse and fill one cuvette (about ¾ full) with the blank (solution 1 from Table 1). Rinse and fill (about ¾ full) a second cuvette with the most dilute of the standard calibration solutions (solution 2 from Table 1). **Gently blot** (don't scratch the sides of the cuvette) off any drips on the outside of the cuvette with a Kimwipe.

   d. Place the cuvette containing the blank in the spectrometer so that one of the clear sides is aligned with the white arrow at the top of the cuvette slot. Click: "Finish Calibration" ; OK. Remove the cuvette from the spectrometer but keep it handy.

6. Determine the Wavelength of Maximum Absorbance

    a. Place the cuvette containing the most dilute standard calibration solution in the spectrometer. Click: Experiment ; Data Collection ; Full Spectrum ; Done. Click the rainbow icon labeled "Absorbance=…" in the upper left hand corner of the window. Change the "Wavelength Range" to 380-750nm. Close this box by clicking the "x" in the upper right hand corner of the window.

    b. Click the small green triangle in the toolbar labeled "Collect". After the line graph appears on the screen, click the small red square in the toolbar labeled "Stop".

    c. To automatically store the maximum wavelength go to the toolbar and select: Experiment ; Store Latest Run.

7. Generate the Calibration Curve

    a. In the toolbar click: Experiment ; Data Collection ; choose Events with Entry in the Mode box. Highlight the word Event in the Column Name box and replace it with Concentration. Put M in the Units box. Clear "Short Name". OK. You are ready to begin collecting data. Remove the cuvette from the spectrometer but keep it handy.

    b. Place the cuvette containing the blank back in the spectrometer. Click the begin data collection button (triangle) in the toolbar labeled "Collect". When the absorbance reading stabilizes, click the KEEP button located in the toolbar just to the right of the Red Stop Button. Type in the concentration of the solution that is in the cuvette (don't include units). OK.

    c. Working in order of most dilute (you already have a cuvette containing this solution) to most concentrated of the standard calibration solutions (not the solutions from Table 2), rinse and then fill a cuvette with the solution that will be analyzed. Place the cuvette in the spectrometer. When the absorbance reading stabilizes, click the KEEP button (be careful that you **don't** accidentally click the stop data collection button (square)), and enter the concentration of the analyzed solution. Repeat until the absorbance of each solution has been determined.

        Pour your samples back into the appropriate TT after you have measured each absorbance and discard them in the waste container only after you have acceptable data.

    d. When the absorbance of all standard calibration solutions (not the solutions from Table 2) has been measured, click the stop data collection button (square) located in the toolbar.

    e. To determine the equation of the line for your calibration curve click: Analyze ; Linear Fit. A box should appear with the equation and a correlation.

        To receive full credit for this lab your calibration curve must be a good, straight-line graph, with a correlation coefficient of 99% or better (Corr: on the screen reads 0.9900 or greater). You should repeat the experiment until you get this proficiency.
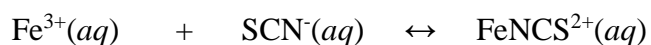
        Work carefully. If you need to repeat the experiment, you still must be done with the write up and post-lab questions before the end of the lab period.

8. Each lab report must have a Logger Pro generated printout of the calibration curve attached to it. The printout must show the graph, the information needed to generate the equation (slope and intercept) for the line, and the correlation reading. To do this click: File ; Print. Uncheck the "Print Visible Spectrum on Wavelength Graphs" and change the "orientation" to landscape under properties. Be sure that the names of all lab partners are entered in the "Name" section and that the date box is checked.

9. Measure the absorbance of each solution in Table 2 and enter the absorbance in the appropriate column in Table 2. Remember to first rinse the cuvette with the solution that will be analyzed.

10. Use the calibration curve and the equation of the line to determine the equilibrium concentration of $FeNCS^{2+}$. Enter this $FeNCS^{2+}$ concentration in Table 2 as well as in the appropriate places in the next five tables (Table 2: A-E).
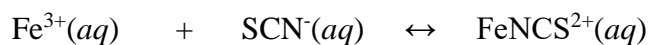
EXAMPLES OF DATA AND ANALYSIS SHEETS
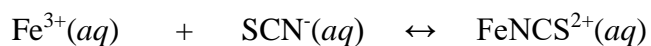
Table 2.

Solution	0.0020 M NaSCN (in 0.25 M HNO₃)	0.0020 M Fe(NO₃)₃ (in 0.25 M HNO₃)	0.25 M HNO₃	Absorbance	[FeNCS²⁺]
A	1 mL	5 mL	4 mL		
B	2 mL	5 mL	3 mL		
C	3 mL	5 mL	2 mL		
D	4 mL	5 mL	1 mL		
E	5 mL	5 mL	0 mL		

$$Fe^{3+}(aq) \quad + \quad SCN^-(aq) \quad \leftrightarrow \quad FeNCS^{2+}(aq)$$

Table 2: A	Fe^{3+}	SCN^-	$FeNCS^{2+}$
Initial Moles			
Change			
Initial + Change			
Equilibrium Moles			
Equilibrium Molar Concentration			
Numerical Value of K_c			

$$Fe^{3+}(aq) \quad + \quad SCN^-(aq) \quad \leftrightarrow \quad FeNCS^{2+}(aq)$$

Table 2: B	Fe^{3+}	SCN^-	$FeNCS^{2+}$
Initial Moles			
Change			
Initial + Change			
Equilibrium Moles			
Equilibrium Molar Concentration			
Numerical Value of K_c			

$$Fe^{3+}(aq) \quad + \quad SCN^-(aq) \quad \leftrightarrow \quad FeNCS^{2+}(aq)$$

Table 2: C	Fe^{3+}	SCN$^-$	FeNCS^{2+}
Initial Moles			
Change			
Initial + Change			
Equilibrium Moles			
Equilibrium Molar Concentration			
Numerical Value of K$_c$			

$$Fe^{3+}(aq) \quad + \quad SCN^-(aq) \quad \leftrightarrow \quad FeNCS^{2+}(aq)$$

Table 2: D	Fe^{3+}	SCN$^-$	FeNCS^{2+}
Initial Moles			
Change			
Initial + Change			
Equilibrium Moles			
Equilibrium Molar Concentration			
Numerical Value of K$_c$			

$$Fe^{3+}(aq) \quad + \quad SCN^-(aq) \quad \leftrightarrow \quad FeNCS^{2+}(aq)$$

Table 2: E	Fe^{3+}	SCN$^-$	FeNCS^{2+}
Initial Moles			
Change			
Initial + Change			
Equilibrium Moles			
Equilibrium Molar Concentration			
Numerical Value of K$_c$			