Worksheet #9

Name: **Period:** **Seat#:**

Introduction

The equilibrium state of a chemical reaction can be characterized by quantitatively defining its equilibrium constant, K_{eq}. In this experiment, you will determine the value of K_{eq} for the reaction between iron (III) ions and thiocyanate ions, SCN^-.

$$Fe^{3+}_{(aq)} + SCN^{-}_{(aq)} \leftrightarrow FeSCN^{2+}_{(aq)}$$

When you mix amounts of Fe^{3+} and SCN^-, a reaction occurs to produce $FeSCN^{2+}$, but not all of the reactants react. Thus, your beaker (or flask or cauldron) will contain some of each of these three species, which is your equilibrium system. To learn more about the system, we need to figure out a way to count the number of different ions in the reaction mixture. That is the major objective of this experiment, and to achieve this objective you will take advantage of something about $FeSCN^{2+}$ – in aqueous solution it has a reddish color. The two reactants, Fe^{3+} and SCN^-, are essentially colorless in solution, thus the red color you will see when you conduct the reaction is produced by the $FeSCN^{2+}$ ions.

One of the more important numbers that help us understand an equilibrium system is called the equilibrium constant, K_{eq}. For the reaction between Fe^{3+} and SCN^-, the K_{eq} is defined by the equation

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

To find the value of K_{eq} at a given temperature, it is necessary to determine the molar concentration of each of the three species in solution at equilibrium. You will determine the concentrations by using a Vernier Colorimeter or Spectrometer to measure the amount of light of a specific wavelength that passes through a sample of the equilibrium mixtures. The amount of light absorbed by a colored solution is proportional to its concentration. The red $FeSCN^{2+}$ solution absorbs blue light, thus the Colorimeter users will be instructed to use the 470 nm (blue) LED. Spectrometer users will determine an appropriate wavelength based on the absorbance spectrum of the solution. The wavelength will be close to, but not exactly, 470 nm.

In order to successfully evaluate this equilibrium system, it is necessary to conduct two separate tests. In Part I of the experiment, you will prepare a series of standard solutions of $FeSCN^{2+}$ from solutions of varying concentrations of SCN^- and constant concentrations of H^+ and Fe^{3+} that are in stoichiometric excess. The excess of H^+ ions will ensure that Fe^{3+} engages in no side reactions (to form $FeOH^{2+}$, for example) which could interfere with your measurements. In an excess of Fe^{3+} ions, the SCN^- ions will be the limiting reagent, thus all of the SCN^- will form $FeSCN^{2+}$ ions. The $FeSCN^{2+}$ complex forms slowly, taking at least one minute for the color to develop. It is best to take absorbance readings after a specific length of time has passed, between two and four minutes after preparing the equilibrium mixture. Do not wait much longer than five minutes to take readings, however, because the mixture is light sensitive and the $FeSCN^{2+}$ ions will slowly decompose.

In Part II of the experiment, you will prepare a new series of solutions that have varied concentrations of the SCN^- ions and constant concentrations of H^+ ions and Fe^{3+} ions. You will use the results of this test to accurately evaluate the equilibrium concentrations of each species and calculate the K_{eq} of the reaction.

Objectives

In this experiment, you will

- Prepare and test standard solutions of $FeSCN^{2+}$ in equilibrium.
- Determine the molar concentrations of the ions present in an equilibrium system.
- Determine the value of the equilibrium constant, K_{eq}, for the reaction.

Materials

Chemicals

- 0.200 M Iron (III), Fe^{3+}, solution in 1.0 M HNO_3
- 0.0020 M Iron (III), Fe^{3+}, solution in 1.0 M HNO_3
- 0.00200 M Thiocyanate, SCN^-, solution
- Distilled water

Equipment

- Computer
- Vernier computer interface*
- Logger Pro
- Colorimeter or Spectrometer
- 10 mL graduated cylinder x4
- 50 mL graduated cylinder
- Small beakers (100-250 mL) x 7
- Plastic cuvettes
- Kim Wipes
- Plastic Beral pipets - several
- *no interface required if using a Spectrometer

SAFETY PRECAUTIONS
DANGER: *Iron (III) nitrate solution,* $Fe(NO_3)_3 \cdot 9H_2O$: *Causes severe skin burns and eye damage. Do not breathe mist, vapors, or spray*. **WARNING**: *Potassium thiocyanate solution,* KSCN: *Causes eye irritation and mild skin irritation*.

Prelab Questions (Part of your Prelab Assignment)

For the solutions that you will prepare in Step 2 of Part I below, calculate the [$FeSCN^{2+}$]. Presume that all of the SCN^- ions react. In Part I of the experiment, mol of SCN^- = mol of $FeSCN^{2+}$. Record these values in the following table:

Beaker #	[$FeSCN^{2+}$]
1	
2	
3	
4	

Procedure

Part I Prepare and Test Standard Solutions

1) Obtain and wear goggles.

2) Label four small beakers 1–4. Obtain small volumes of 0.200 M $Fe(NO_3)_3$, 0.0020 M SCN^-, and distilled water. Prepare four solutions according to the chart below. Use graduated cylinders to measure the solutions. Mix each solution thoroughly. Record the temperature of one of the solutions as the temperature for the equilibrium constant, K_{eq}. **DANGER:** *Iron (III) nitrate solution,* $Fe(NO_3)_3 \cdot 9H_2O$: *Causes severe skin burns and eye damage. Do not breathe mist, vapors, or spray*. **WARNING**: *Potassium thiocyanate solution,* KSCN: *Causes eye irritation and mild skin irritation*.

Important: The mixtures you will prepare are light sensitive. You need to measure the absorbance of these four mixtures **within 2–5 minutes** of preparing them.

Beaker	0.200 M $Fe(NO_3)_3$ (mL)	0.0020 M SCN^- (mL)	H_2O (mL)
1	5.0	4.0	41.0
2	5.0	3.0	42.0
3	5.0	2.0	43.0
4	5.0	1.0	44.0

3) Prepare a *blank* by filling a cuvette 3/4 full with 0.200 M $Fe(NO_3)_3$. To correctly use cuvettes, remember:
- Wipe the outside of each cuvette with a lint-free tissue.
- Handle cuvettes only by the top edge of the ribbed sides.
- Dislodge any bubbles by gently tapping the cuvette on a hard surface.
- Always position the cuvette so the light passes through the clear sides.

Spectrometer Users Only (Colorimeter users proceed to the Colorimeter section)

4) Use a USB cable to connect the Spectrometer to the computer. Choose New from the File menu.

5) To calibrate the Spectrometer, place the blank cuvette into the cuvette slot of the Spectrometer, choose Calibrate ► Spectrometer from the Experiment menu. Wait for the Spectrometer to warm up, then click [OK].

6) Determine the optimum wavelength for the equilibrium mixture and set up the mode of data collection.

- **a)** Empty the 0.200 M $Fe(NO_3)_3$ from the blank cuvette. Using the solution in Beaker 1, rinse the cuvette twice with ~1 mL amounts and then fill it 3/4 full. Wipe the outside with a tissue and place the cuvette in the Spectrometer.
- **b)** Click [▶ Collect]. The absorbance *vs*. wavelength spectrum will be displayed. Note that one area of the graph contains a peak absorbance. Click [■ Stop].
- **c)** To save your graph of absorbance *vs*. wavelength, select Store Latest Run from the Experiment menu.
- **d)** Click the Configure Spectrometer Data Collection icon, , on the toolbar. A dialog box will appear.
- **e)** Select Absorbance *vs*. Concentration under Set Collection Mode. The wavelength of maximum absorbance (λ max) is automatically identified. The λ max should be 400–480 nm. Click [OK].
- **f)** Proceed directly to Step 7.

Colorimeter Users Only

4) Connect the Colorimeter to the computer interface. Open the file "10 Equilibrium" from the *Advanced Chemistry with Vernier* folder of Logger *Pro*.

5) Open the Colorimeter lid, insert the blank, and close the lid.

6) Calibrate the Colorimeter and prepare to test the standard solutions.

- **a)** Press the < or > button on the Colorimeter to select the 470 nm wavelength.
- **b)** Press the CAL button until the red LED begins to flash and then release the CAL button.
- **c)** When the LED stops flashing, the calibration is complete.
- **d)** Empty the water from the blank cuvette. Using the solution in Beaker 1, rinse the cuvette twice with ~1 mL amounts and then fill it 3/4 full. Wipe the outside with a tissue, place it in the Colorimeter, and close lid.

Both Colorimeter and Spectrometer Users

7) Collect absorbance-concentration data for the four standard equilibrium mixtures.
- **a)** Leave the cuvette, containing the Beaker 1 mixture, in the device (Colorimeter or Spectrometer).
- **b)** Click [▶ Collect]. After the absorbance reading stabilizes, click [Keep], type the concentration of $FeSCN^{2+}$ (from your pre-lab calculations) in the edit box, and click [OK].
- **c)** Discard the cuvette contents as directed. Rinse and fill the cuvette with the solution in Beaker 2 and place it in the device. After the reading stabilizes, click [Keep], type the concentration of $FeSCN^{2+}$ in the edit box, and click [OK].
- **d)** Repeat Part c of this step to measure the absorbance of the solutions in Beakers 3 and 4.
- **e)** Click [■ Stop] after you have finished collecting data from the four beakers of reaction mixtures. Click Examine, , and write down the absorbance values in your data table.

8) Click Linear Fit, . A best-fit line (linear regression) equation will be plotted for your data. Write down the equation in your Data Table.

IMPORTANT: Don't change anything in Logger *Pro*. You will use the best-fit line equation in Part II.

Part II Prepare and Test Equilibrium Systems

9) Label three new small beakers A–C. Prepare the solutions according to the chart below. Use 10.0 mL graduated cylinders to measure the solutions. Mix each solution thoroughly. **Note**: You are using 0.0020 M $Fe(NO_3)_3$ in this test.

WARNING: *Iron (III) nitrate solution,* $Fe(NO_3)_3 \cdot 9H_2O$: *Causes skin/eye irritation. Do not breathe mist/vapors/ spray*.

Beaker #	0.0020 M $Fe(NO_3)_3$ (mL)	0.0020 M SCN^- (mL)	H_2O (mL)
A	3.00	3.00	4.00
B	3.00	4.00	3.00
C	3.00	5.00	2.00

Calculating Equilibrium Concentrations

10) Collect absorbance-concentration data for the three beakers of equilibrium mixtures.

a) Using the solution in Beaker A, rinse the cuvette twice with ~1 mL amounts and then fill it 3/4 full. Wipe the outside with a tissue and place the cuvette in the device (Spectrometer or Colorimeter.)

b) Write down, in your data table, the absorbance of the sample in Beaker A.

c) Open the Analyze menu and choose Interpolate. Trace along the best-fit line equation to find the $FeSCN^{2+}$ concentration for the sample in Beaker A. Write down the concentration in your data table.

d) Discard the cuvette contents as directed. Rinse and fill the cuvette with the solution in Beaker B and place it in the device. After the reading stabilizes, write down the absorbance in your data table and use the Interpolate function to determine the concentration of the sample.

e) Repeat Step d for the mixtures in Beaker C.

Disposal and Cleanup

Your teacher will provide disposal and cleanup instructions.

Data Table

Part I

Temperature: ________°C

Beaker	$[FeSCN^{2+}]$	Absorbance
1		
2		
3		
4		

Linear regression equation	

Part II

Beaker	Absorbance	$[FeSCN^{2+}]$ at equilibrium
A		
B		
C		

A common method that is used to organize and calculate the concentrations of the species in an equilibrium system is colloquially known as an I.C.E. chart. "I.C.E" stands for *I*nitial concentration, *C*hange in concentration, and the *E*quilibrium concentration. The initial concentrations of the Fe^{3+} and the SCN^{-} ions can be calculated from the mixing chart in Part II, Step 10. You have already determined the equilibrium concentration of the $FeSCN^{2+}$ ions by completing the analysis in Part II. The rest is a little bit of math.

BEAKER A

	Fe^{3+}	SCN^{-}	$FeSCN^{2+}$
Initial			0.00
Change			
Equilibrium			

BEAKER B

	Fe^{3+}	SCN^{-}	$FeSCN^{2+}$
Initial			0.00
Change			
Equilibrium			

BEAKER C

	Fe^{3+}	SCN^{-}	$FeSCN^{2+}$
Initial			0.00
Change			
Equilibrium			

Calculations

Record all values into your Data Table

1. (Part II) Use your data to determine the $[Fe^{3+}]$, $[SCN^{-}]$, and $[FeSCN^{2+}]$ at equilibrium for each of the mixtures that you prepared in Part II. Complete the table below and give an example of your calculations.

	A	B	C
$[FeSCN^{2+}]$			
$[Fe^{3+}]$			
$[SCN^{-}]$			

2. Calculate the value of K_{eq} for the reaction. Explain how you used the data to calculate K_{eq}.