Introduction

This experiment will give you an opportunity to determine the equilibrium constant for the formation of Fe(SCN)$^{2+}$. The experiment will require you to use Le Châtelier's principle.

When the reaction between Fe$^{3+}$ and SCN$^{-}$ (thiocyanate) ions in aqueous solution comes to equilibrium, the system will consist of the reactants and Fe(SCN)$^{2+}$. The chemical equation for this reaction is:

$$\text{Fe}^{3+}(aq) + \text{SCN}^{-}(aq) \rightleftharpoons \text{Fe(SCN)}^{2+}(aq)$$

The product is a complex ion that has a coordinate covalent bond between the iron and an atom (probably the S atom) from the thiocyanate anion. The color of this complex ion is so intense that thiocyanate ions can be used to detect very small quantities of Fe$^{3+}$.

The objective of this experiment is to determine the equilibrium constant for this reaction. The equilibrium constant is given by the expression

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

where the concentrations of the substances are those at equilibrium. If these concentrations are measured or inferred, $K$ can be easily calculated.

Since the reactants are essentially colorless, while the complex ion is deeply colored, you will use a spectrophotometer to monitor the absorbance (at 447 nm) due to the complex ion without interference from the reactants.

You will plot a graph of known concentrations of FeSCN$^{2+}$ versus absorbance. FeSCN$^{2+}$ does not exist in the solid form, therefore you will need to push the reaction far to the right by adding a large excess of SCN$^{-}$ to the reaction. This will use up all of the Fe$^{3+}$ ions. The concentration of FeSCN$^{2+}$ formed as product will be the same as the initial concentration of Fe$^{3+}$. You will use five different concentrations of Fe$^{3+}$ to obtain five known concentrations of FeSCN$^{2+}$. You will find the absorbance for each of these solutions using the spectrophotometer, and then graph your data.

You will prepare ten solutions using varying amounts of Fe$^{3+}$ and SCN$^{-}$ and measure the absorbance (at 447 nm) of each with the spectrophotometer. Using the graph that you prepared, you will determine the concentration of FeSCN$^{2+}$ present in each of the ten equilibrium solutions. With this data, you will be able to calculate the equilibrium constant, $K$.

Materials & Equipment

KSCN solution, 1M

100 mL Volumetric flask
Determination of Equilibrium Constants

KSCN solution, 0.0025M
Fe(NO$_3$)$_3$ solution, 0.0025M (in 0.1M HNO$_3$)
HNO$_3$ solution, 0.1M
Rubber stoppers for 18 x 150 mm test tubes
Spectrophotometer

Procedure

1. Preparation of dilute Fe(NO$_3$)$_3$ solution
   (a) Transfer 4.00 mL of 0.0025M Fe(NO$_3$)$_3$ (in 0.1M HNO$_3$) to a 100 mL volumetric flask. Carefully add distilled water to the 100 mL mark. Mix the solution thoroughly. This solution will now be known as the dilute iron (III) nitrate solution.

2. Preparation of solutions of known [Fe(SCN)$_2^+$]
   (a) Obtain six 18x150 mm test tubes and matching rubber stoppers. Wash, rinse and dry them. Label the tubes 1 through 5, and leave the 6th blank.
   (b) Rinse and fill a buret several times with the dilute Fe(NO$_3$)$_3$ solution prepared in Step 1(a). Using Table 1 as a guide, add the specified amount of the dilute Fe(NO$_3$)$_3$ solution to each of the numbered test tubes.
   (c) Rinse and fill a buret with 1M KSCN solution. Using Table 1 as a guide, add the specified amount of 1M KSCN into each of your five test tubes.
   (d) Rinse and fill a buret with 0.1M HNO$_3$. Add the correct amount of nitric acid solution to each test tube (according to Table 1). The volumes of each of the solutions in the test tubes should now be equal. Insert the rubber stoppers and mix the contents of each tube thoroughly.

3. Measuring absorbance with the spectrophotometer
   (a) Zero your spectrophotometer.
   (b) Set 100% transmittance using distilled water in your cuvette.
   (c) Transfer the solutions, one at a time, to your cuvette. (Be careful to rinse the cuvette with each new concentration of solution before adding it). Measure and record the %T (at 447 nm) of each solution in Table 1. Calculate the absorbance for each solution, using the equation:

\[
\text{Absorbance} = \log \left( \frac{100}{\%T} \right)
\]

and record your results in Table 1.

4. Preparation of solutions using 0.0025M reagents
   (a) Wash, dry, and label ten test tubes and stoppers (1-10).
   (b) Prepare the ten solutions shown in Table 2. Use a properly rinsed and filled buret for each addition. It is easiest to add the Fe(NO$_3$)$_3$ to each of the ten test tubes, add the KSCN to each, and finally add the HNO$_3$.
   (c) After thoroughly mixing these solutions, transfer them to your cuvette as you did in Step 3(c). Measure the %T (at 447 nm). Record your results in Table 2. Calculate the absorbance for each solution and record your results in Table 2.

5. Cleanup
   Clean and rinse your burets, cuvette, and test tubes with distilled water. When you are finished, clean your lab area and return your cuvette to your lab assistant before being signed out.
### Table 1

<table>
<thead>
<tr>
<th>Test Tube#</th>
<th>Dilute Fe$^{3+}$ (mL)</th>
<th>1M SCN$^-$ (mL)</th>
<th>0.1M HNO$_3$ (mL)</th>
<th>%T At 447 nm</th>
<th>Absorbance</th>
<th>[FeSCN$^{2+}$]</th>
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### Table 2

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<th>Test Tube#</th>
<th>0.0025M Fe(NO$_3$)$_3$ (mL)</th>
<th>0.0025M KSCN (mL)</th>
<th>0.1M HNO$_3$ (mL)</th>
<th>%T At 447 nm</th>
<th>Absorbance</th>
<th>(calculate based on graph) [FeSCN$^{2+}$] eq</th>
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<th>[Fe$^{3+}$] initial</th>
<th>[SCN$^{-}$] initial</th>
<th>(from Table 2)</th>
<th>[FeSCN$^{2+}$] eq</th>
<th>[Fe$^{3+}$] eq</th>
<th>[SCN$^{-}$] eq</th>
<th>K</th>
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#### Calculations

For the calculations of this experiment, please use a spreadsheet program, such as MS Excel, to help streamline and simplify the many redundant and inter-related calculations involved. This will save you considerable amounts of time and effort, particular if you make a mistake in one of the early calculations.

**Part 1**

1. Calculate the concentration of the dilute Fe(NO$_3$)$_3$, as this is also the concentration of the product, FeSCN$^{2+}$.
   
   The formula for calculating the concentration is:
   
   \[
   \frac{(0.0025 \text{ M Fe}^{3+})(4.0 \text{ mL})}{(100.0 \text{ mL})} = \text{concentration of the diluted Fe}^{3+}
   \]

   Now calculate the initial concentration of the Fe$^{3+}$ after mixing it with the KSCN and the HNO$_3$.

   \[
   \frac{([\text{diluted Fe}^{3+}](\text{no. mL of Fe}^{3+} \text{ used})}{(\text{total no. of mL soln})} = [\text{FeSCN}^{2+}]
   \]

   Record the concentrations of product, FeSCN$^{2+}$, on Table 1.

2. Construct a graph of [FeSCN$^{2+}$] versus absorbance. The graph must be at least half a page, and must conform to the graphing guidelines set forth by your instructor. The y
Determination of Equilibrium Constants

axis should have a range of 0.0 to 10.0, and should indicate that these values are “x $10^{-5}$.” The x axis should have a range of 0.00 to 1.00 for absorbance at 447 nm.

Part 2

1. Calculate the initial concentrations of Fe$^{3+}$ and SCN$^{-}$ using the same type of formula as in Part 1. Fill in the appropriate columns in Table 3 with this information.

2. Find the equilibrium concentrations of FeSCN$^{2+}$ by using the graph you prepared and the absorbancies you listed in Table 2 (See Note 1). Copy the data to Table 3.

3. Calculate the equilibrium concentrations of Fe$^{3+}$ and SCN$^{-}$ by subtracting the equilibrium concentrations of FeSCN$^{2+}$ from the initial concentrations of Fe$^{3+}$ and SCN$^{-}$. Fill in the appropriate columns on Table 3.

4. Calculate the equilibrium constant, K, by using the formula

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

Do this for each trial and then determine the average value for the equilibrium constant.

Note 1

When finding the equilibrium concentrations of FeSCN$^{2+}$, use the equation

$$y = mx + b$$

The values of m and b will be calculated by the graphing software, and the absorbance (depending on which axis you have it plotted) will be either y or x, leaving the other variable to solve for.

Acknowledgement*

This lab was developed at Wabash College in Crawfordsville, Indiana at the Wabash College High School Teachers Program in Quantitative Biology. This program was sponsored by the Howard Hughes Medical Institute through a grant from HHMI to Wabash College. [This lab] was written by Barbara Gabet of Snider High School, Fort Wayne, Indiana.

*Alterations have been made to the overall composition and design of the lab.