Experiment 25: Determination of K_c for a Chemical Reaction

*This experiment was adapted from **Ross S. Nord**, Chemistry Department, Eastern Michigan University.*

Purpose: To determine the equilibrium constant for the reaction between iron(III) and thiocyanate, and to study the equilibrium system of this chemical reaction.

Background: Iron(III) and thiocyanate react to form an equilibrium mixture with the product formed; the iron-thiocyanate complex ion. The net-ionic equation is:

$$Fe^{3+}_{(aq)} + SCN^{1-}_{(aq)} \rightleftharpoons FeNCS^{2+}_{(aq)}$$

The iron(III) solution is slightly pale-yellow in color, the thiocyanate solution in colorless, and the complex ion product is an orange-red color. The colored product will allow for the use of a spectrophotometer to quantitate the amount of product made. Using the data from the spectrophotometer, along with concentrations of iron(III) and thiocyanate, we will study this equilibrium system.

When a reaction reaches equilibrium, not all of the reactants became product. A mixture of reactants and products exist. The equilibrium constant is calculated as the ratio of the concentration of product(s) divided by the concentration of reactant(s). This is the simplified version of the math. The more complicated version involves calculations with activity coefficients and ionic strength due to the fact that this equilibrium system involves ionic species. Overall, we can say that it is typical for the math to be simplified for the calculation of the equilibrium constant.

$$Kc = \frac{[FeNCS^{2+}]}{([Fe^{3+}] \times [SCN^{1-}])}$$

This experiment consists of three parts:

Part 1: Determine the λ_{max} for the $FeNCS^{2+}$ ion. Since the solution is an orange-reddish color, wavelengths in the 400 nm to 500 nm range are being absorbed.

Part 2: The reaction between the iron(III) and thiocyanate will be studied as an equilibrium, with the ion concentration as low as possible. Reasonable amounts of each reactant will be combined to allow the equilibrium mixture to form.

Part 3: The reaction between the iron(III) and thiocyanate will be studied as an equilibrium, with the ion concentration increased by the addition of 0.10 M KNO_3. The amount of each reactant will be the same as in Part 2.

Chemicals *(Use caution with each of these solutions.)*
0.0020 M $Fe(NO_3)_3$ in 0.0050 M HNO_3
0.0020 M NaSCN in DI water
0.10 M KNO_3
Deionized water

Equipment
1.0 mL pipet (1), 10.0 mL pipet (2), pipettor (2), beakers (3)
Small test tubes
Parafilm
Spectrophotometer

Procedure
Part 1: Determining λ_{max} for $FeNCS^{2+}$

a) Combine 1.50 mL of Fe^{3+} with 2.00 mL of SCN^{1-} in a small test tube.
b) Cover the test tube with Parafilm. Secure your thumb over the Parafilm and invert the test tube to mix. Invert to mix three times. This is the equilibrium mixture.
c) Place a test tube of Fe^{3+} solution in the sample compartment of the spectrophotometer and zero the instrument at 400 nm. (This is the blank solution.)
d) Place the test tube with the equilibrium mixture in the sample compartment and record the absorbance of the mixture. Remove this solution from the sample compartment.
e) Increase the wavelength by 5 nm.
f) Repeat steps c – e until you have reached 500 nm.
g) Graph the absorbance of light on the Y-axis and the wavelength (nm) on the X-axis, from 400 nm to 500 nm. Determine the best wavelength to use for the analysis of $FeNCS^{2+}$.

Part 2: Observe the Equilibrium Mixture with Low Ionic Strength

a) Prepare a series of equilibrium mixtures, each in its own small test tube. Use the volumes of each reactant listed in Table 1.
b) Cover each test tube with Parafilm. Secure the film with your thumb and invert to mix three times.
c) Set the spectrophotometer wavelength to λ_{max}. Use the Fe^{3+} solution as your blank and zero the spectrophotometer.
d) Measure the absorbance of each equilibrium mixture.
e) Add 1.5 mL of DI water to each test tube, and invert to mix three times.
f) Measure the absorbance of each equilibrium mixture.
g) Add 1.0 mL of DI water to each test tube, and invert to mix three times.
h) Measure the absorbance of each equilibrium mixture.

Table 1 Solution Preparation for Part 2

Solution Number	Volume of Fe^{3+}, mL	Volume of SCN^{1-}, mL
Blank	4	0
1	0.50	3.00
2	1.00	2.50
3	1.50	2.00
4	2.00	1.50
5	2.50	1.00
6	3.00	0.50

Part 3: Observe the Equilibrium Mixture with High Ionic Strength

a) Prepare a series of equilibrium mixtures, each in its own small test tube. Use the volumes of each reactant listed in Table 1 (same as in Part 1).
b) Cover each test tube with Parafilm. Secure the film with your thumb and invert to mix three times.
c) Set the spectrophotometer wavelength to λ_{max}. Use the Fe^{3+} solution as your blank and zero the spectrophotometer.
d) Measure the absorbance of each equilibrium mixture.
e) Add 1.5 mL of 0.10 M KNO_3 to each test tube, and invert to mix three times.
f) Measure the absorbance of each equilibrium mixture.
g) Add 1.0 mL of 0.10 M KNO_3 to each test tube, and invert to mix three times.
h) Measure the absorbance of each equilibrium mixture.

Calculations

Use Excel or Google Sheets for the calculations. This will save a lot of time and provide experience with programming spreadsheets. There is a video in your Blackboard shell that shows you how to make the Excel / Google Sheets file for this experiment.

You will have to calculate the following:

a) The initial concentration of iron(III).
b) The initial concentration of thiocyanate.
c) The concentration of $FeNCS^{2+}$ made (this is the equilibrium concentration). Remember Beer's Law, A=abc. Make the spreadsheet solve for c, the concentration. You know the absorbance, A. Use 5800 $M^{-1}\cdot cm^{-1}$ for the extinction coefficient a, and use 1 cm for the path length b.
d) The equilibrium concentration of iron(III).
e) The equilibrium concentration of thiocyanate.
f) The value of the equilibrium constant, using equilibrium concentrations.

All of these calculations are shown in the prelab PowerPoint file and in the video.

Conclusion

When you write your conclusion, consider these topics:

a) Was the equilibrium constant a constant within each table, considering experimental error?
b) Was the equilibrium constant a constant after the dilution of water, considering experimental error?
c) Was the equilibrium constant affected by the addition of KNO_3, more so than the addition of DI water?
d) The preparation of the original solutions was the same in Part 2 and Part 3. How do those equilibrium constant values compare?
e) Did you notice any patterns with the data?

There is graph paper on the next page for Part 1, finding λ_{max}.