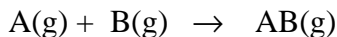


EXPERIMENT 6: Photometric Determination of an Equilibrium Constant

The following preparatory questions should be answered before coming to class. They are intended to introduce you to several ideas important to aspects of the experiment. You must turn-in your work to your teaching assistant before you will be allowed to begin the experiment. Be sure to bring a calculator and paper to laboratory.

1. The equilibrium constant, K_c , for the reaction



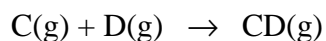
is 130 at 25 °C. If 8.750×10^{-2} mol of A and 6.312×10^{-5} mol of B are introduced into a 1.00 liter vessel at 25 °C, calculate the equilibrium concentration of all species.

2. Answer the following questions regarding problem #1:
 - a) How does the amount of B reacting compare to initial amount of B?

 - b) How does the amount of AB at equilibrium compare to the initial amount of B?

 - c) What is it about the initial conditions that might explain your answer in 2 b)?

3. The equilibrium constant, K_C , for the reaction



is unknown at 25 °C. If 9.119×10^{-2} mol of C and 4.672×10^{-5} mol of D are introduced into a 1.00 liter vessel at 25 °C, calculate the equilibrium concentration of CD.

Discuss any assumptions you made in arriving at your answer.

4. a) This experiment is separated into parts I and II. What are the $[Fe^{3+}]$ and $[SCN^-]$ before mixing in each part?

Part I	Part II
$[Fe^{3+}] =$	$[Fe^{3+}] =$
$[SCN^-] =$	$[SCN^-] =$

- b) Briefly, explain why the concentrations of the Fe^{3+} and SCN^- are different in the two parts of the experiment.
- c) How are you going to keep from mixing the wrong combinations when you are doing the experiment?

EXPERIMENT 6: Photometric Determination of an Equilibrium Constant

IMPORTANT: This experiment uses low toxicity materials which can go down the drain once the solutions are neutralized. Instructions for neutralizing the solutions are at the end of the Procedure, p. 10. Please set aside your 600 mL beaker as a waste container. Collect all solutions in that beaker.

The purpose of this experiment is to determine the equilibrium constant for a chemical reaction.

To begin the experiment obtain 10 mL samples of $\text{Fe}(\text{NO}_3)_3$ and KSCN from the reagent bottles marked "stock solution" from the center lab bench. Mix the two solutions and record your observations in your Laboratory Notebook. Be sure to note properties before and after solutions are mixed. Include the molecular, ionic and net ionic equation that best describes the reaction that occurred when the two solutions were mixed. In the net ionic equation indicate the color of all species.

The equilibrium constant, K_c , can be calculated if the equilibrium concentration of all species, in the equilibrium expression above, are known.

In the experiment, known amounts of the reactants Fe^{3+} and SCN^- (thiocyanate ion) will be allowed to react to form the product FeSCN^{2+} . If there were a method of determining the amount of FeSCN^{2+} at equilibrium it would be possible to stoichiometrically calculate the equilibrium concentration of the reactants. Substituting into the equilibrium expression above would yield the equilibrium constant.

Fortunately, because FeSCN^{2+} is colored, a light measuring device called a Spectronic 20 can be used to determine, quantitatively, the concentration of FeSCN^{2+} formed at equilibrium. The Spectronic 20 measured the amount of light absorbed by the colored species, FeSCN^{2+} . Mathematically the amount of light absorbed by the FeSCN^{2+} is directly proportional to the concentration of FeSCN^{2+} , according to the equation:

$$\text{Absorbance (A)} = a \cdot b \cdot [\text{FeSCN}^{2+}]$$

where 'a' and 'b' are constants unique to the colored compound and the experiment.

The experiment consists of two parts. In Part I the absorbance of samples of known concentration of FeSCN^{2+} will be measured. This data will be plotted on a sheet of graph paper to obtain a calibration line. In Part II the absorbance of samples containing an unknown concentration of FeSCN^{2+} will be measured.

Question:

- 1) Write the equilibrium constant expression for the net ionic reaction.
- 2) In your own words briefly, discuss the importance of Part I of the experiment and how the results of Part I will be used in Part II.

Experimental Procedure: Work in pairs

Checkout: 1 – 10 mL Mohr pipet and pipet pump
 (per pair) 5 – 18 x 150 mm test tubes
 5 – Spectronic 20 test tubes (note: these Spec 20 test tubes are very expensive.)

Please return the test tubes and the Spec 20 tubes **clean and dry!** Thanks

Part I. Calibration Line (Note: The concentration of reagents vary in Part I and Part II)A. Preparing Solutions:

Place the five 18 x 150 mm test tubes in a test tube rack and clearly label them A through E. Obtain 60 mL of the 0.200 M $\text{Fe}(\text{NO}_3)_3$ solution in a 100 mL beaker, 20 mL of 9.00×10^{-4} M KSCN in a 50 mL beaker and 40 mL of 0.500 M HNO_3 in another 50 mL beaker. Write the chemical equations which indicate the ions that are in these three solutions.

When solutions A – E are prepared, extreme care must be exercised to insure that the exact amount of each reagent is added to each test tube. To do this use the Mohr pipets to dispense the specific volume. (**DO NOT MOUTH PIPET.**) Follow your TA's instructions on how to properly use a Mohr pipet. Practice with it before beginning the mixing of solutions A – E. A few minutes of practice could prevent having to repeat the experiment. If at any time during the preparation of the mixtures a solution in a Mohr pipet turns brownish red, then the pipet is contaminated. It must be cleaned and the solution for that test tube prepared again. Suggestion: add one of the reagents to all the test tubes, clean the Mohr pipet and add another reagent to the test tube, etc.

Pipet into each test tube the amount of each reagent specified in Table I. Carefully mix the solution so the color is homogeneous.

Table I

Test Tube	mL of .200 M $\text{Fe}(\text{NO}_3)_3$	mL of 9.00×10^{-4} M KSCN	mL of .500 M HNO_3
A	10.00	5.00	5.00 B
10.00	4.00	6.00	C
10.00	3.00	7.00	D
10.00	2.00	8.00	E
10.00	0.00	10.00	

Solution E is termed the "blank."

B. Calibrating Spectronic 20 and Measuring Sample Absorption of Solutions A–D.

After the Spectronic 20 has warmed up for 30 minutes, adjust the wavelength dial to 480 nanometers (nm). Measure the absorbance (% transmittance) of solutions A – D. Use a Kim-wipe and clean the outside of the Spectronic 20 test tube. Prepare a table and record your data in your Laboratory Notebook.

Using the data in Table I, several calculations must be performed at this point in the experiment. First, the initial concentrations of the reactant ions must be determined. Table I provides the concentrations of the 'stock' solutions of each ion and the volumes in each test tube. Using this data prepare a table of initial and final (equilibrium) concentrations of Fe^{3+} , SCN^- and FeSCN^{2+} in your Laboratory Notebook. Note: Be sure to take into account the dilution effect.

After completing the concentration table, prepare a calibration line using a piece of graph paper or use the graphing software on the Macintosh.

Part II. Determination of K_c **A. Preparing Solutions:**

Clean the test tubes containing solutions A–D and re-label as solutions F through I. Do not throw away Solution E. Fill each test tube with the amounts of each reagent specified in Table IV and carefully mix each solution so the color is homogeneous. As in Part I, be as accurate as possible when using the Mohr pipet to prepare the Solutions F–I.

Table IV

Test Tube	mL of $1.00 \times 10^{-2} \text{ M Fe}^{3+}$	mL of $1.13 \times 10^{-3} \text{ M SCN}^{-}$
F	2.50	2.50
G	2.50	5.00
H	5.00	2.50
I	5.00	1.00

Before measuring the absorbances of Solutions F–I, check the calibration of the Spectronic 20 using the blank (Solution E).

Prepare a data table in your Laboratory Notebook then measure and record the absorbance² and % transmittance of Solutions F–I in the data table below (Table V). Remember to wash the Spectronic 20 test tube with a small amount of the solution to be measured before filling the test tube. Also clean the outside of the test tube using the Kim-wipes.

CLEAN-UP:

1. Fill a 50 mL beaker to the 20 mL mark with solid sodium carbonate, Na_2CO_3 .
2. To neutralize the waste put the waste beaker in the sink and sprinkle solid Na_2CO_3 into the beaker. When the fizzing stops, pour the solution down the drain with running water.
3. Wash your glassware, shake the pipet dry, dry the test tubes and Spec 20 tubes with a paper towel, and return them to the storeroom.
4. Wash and dry your lab bench.
5. Wash your hands before leaving the lab

CALCULATIONS

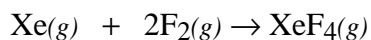
Prepare a table in your Laboratory Notebook which includes the initial and equilibrium concentrations of Fe^{3+} , SCN^{-} and FeSCN^{2+} based on the new reaction conditions. Calculate K_c for the reaction. Be sure to include a clear narrative describing how the data was treated to obtain the equilibrium constant.

² In the event that the % transmittance data is more accurate than the absorbance data, the following equation may be used to convert to % transmittance data to absorbance:

$$A = 2 - \log \%T$$

Post Lab Questions

1. At a certain temperature the reaction



gives a 50% yield, starting with 0.400 mol of Xe and 1.00 mol of F₂ in a 2.00 L vessel. Calculate the equilibrium constant, K_c, for the reaction.

2. In the space below enter the concentrations of Fe³⁺ and SCN⁻ in test tube 'C'.

Fe³⁺ = _____ M

SCN⁻ = _____ M

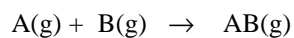
absorbance = _____

When a few drops of Fe³⁺ are added to test tube 'C' the absorbance of the solution does not change significantly. Briefly, explain this observation.

When a few drops of SCN⁻ are added to test tube 'C' the absorbance of the solution does change. Briefly, explain this observation.

3. Consider Problem #1 on page 1 of the experiment. What would be the percentage error in the equilibrium concentration of AB if we assumed the equilibrium concentration of AB was equal to the initial concentration of B rather than the amount calculated?

The equilibrium constant, K_c , for the reaction



is 130 at 25 °C. If 8.750×10^{-2} mol of A and 6.312×10^{-5} mol of B are introduced into a 1.00 liter vessel at 25 °C, calculate the equilibrium concentration of all species.

4. If, while preparing solutions A in Part I, the solution in the pipet turns brownish red, explain what has happened and why it is necessary to repeat the preparation of that test tube.
