

Lab Procedure

Measuring an Equilibrium Constant I: Using Color to Analyze an Equilibrium Mixture

Instructions for preparing for this experiment are provided in the Online Manual. Be sure to read and study that material, along with these procedures, before coming to lab. During the pre-laboratory class, your instructor will explain terms in boldface type. Ask your instructor to explain any aspects of the procedures that you do not understand.

As you work, keep full, legible records of your work and observations in your Laboratory Notebook. Enter data in the tables you prepared before coming to lab (see instructions at the beginning of each Part, below). You will hand in the yellow copies of your lab work with the Report Form.

Procedures

Work in pairs to collect data. Carry out all other work and reporting individually.

Part 1. Obtaining Calibration Data for FeSCN^{2+} Solutions

a) Calibration Solutions

*CAUTION: The $\text{Fe}(\text{NO}_3)_3$ reagents for Parts 1 and 2 are **different: 0.200 M in Part 1, and 0.00200 M in Part 2. Read instructions and reagent labels carefully.***

In your lab notebook, set up a data table in the format shown in Part 1a of the **Report Form**. Enter data and results in your lab notebook as you proceed.

Prepare each calibration solution by pipetting the amounts of **0.200M** $\text{Fe}(\text{NO}_3)_3$ and 2.00×10^{-3} M KSCN shown in Table 1 into a clean 25-mL **volumetric flask**. Increase the solution volume to precisely 25.00 mL by adding 0.1 M HNO_3 until the flask is filled to the mark. Add the last mL with a dropper in order to avoid filling past the mark. Stopper the flask securely and mix completely by inverting and rotating the flask several times. Transfer each solution to a clean, dry flask labeled with the calibration solution number. Rinse the volumetric flask thoroughly with distilled water before preparing the next solution. **Make all five solutions before proceeding to Part 1b.**

Chemistry Department, University of Southern Maine

Lab Procedure

TABLE 1 Calibration Solution #	0.200 M Fe(NO₃)₃ (in 0.1 M HNO ₃), mL	2.00 x 10⁻³ M KSCN (in 0.1 M HNO ₃), mL
C1 (blank)	5.00	0.00
C2	5.00	1.00
C3	5.00	1.50
C4	5.00	2.00
C5	5.00	2.50

b) Calibration Data

Set the wavelength of the spectrophotometer to 460 nm. Set **Mode** to **TRANSMITTANCE**.

With the sample compartment empty (the light to the detector is blocked), use the **Zero** control (**left-hand knob on front**) to set the instrument to 0.00 %T. This corresponds to infinity on the **A** scale, so you must make this setting in transmittance mode.

Obtain a sample cuvette. Make sure it is clean inside and out. Avoid touching the bottom third of the tube, to keep its light path clear. **Rinse the cuvette in**, with small amounts (~1 mL) of Solution C1 (the blank). Then fill the cuvette about two-thirds full. Wipe off the outside of the the cuvette, and insert it into the sample compartment with its orientation mark aligned with the mark in the spectrophotometer. Set **Mode** to **ABSORBANCE**. Use the **Blank** control (**right-hand knob on front**) to set the instrument to 0.000 A. After this point, be careful not to disturb the **Zero** or **Blank** controls.

Return Solution #1 to its flask. **Rinse in** the cuvette with solution #2, wipe it off, insert it into the instrument, and read the absorbance. Repeat for the remaining calibration solutions, recording absorbances to three decimal places in a data table in your lab notebook.

LANGUAGE NOTE: Because you have set the instrument to **A** = 0.000 with solution C1, we say that you have “determined the absorbance of solutions C2 through C5 against C1 as a blank.”

Finally, check to be sure that the instrument is still properly blanked and zeroed, as follows: **rinse in** and fill the cuvette again with Solution C1. Read its absorbance. It should still be very close to 0.000. Remove the cuvette and read transmittance. It should still be close to 0.00. If either reading has changed by more than 5 in the last decimal place, you should repeat Part 1b.

Lab Procedure**c) Beer's-Law Plot**

After lab, use Excel to plot your data as **absorbance** (vertical axis) vs. **concentration** (horizontal axis). Fit a straight line to your data, and obtain the equation of the line and the regression coefficient (R^2). Label the axes of the graph and give it an informative title from which the reader can readily understand the relationship represented on the graph. Make sure the graph meets all syllabus guidelines. Make three copies of the graph. Hand in one with your report. Mount the other two on corresponding white and yellow pages of your lab notebook.

Working in your lab notebook, determine **E** for the FeSCN^{2+} ion.

Part 2. Obtaining Absorbance Data for Equilibrium Mixtures**a) Equilibrium Absorbance Data**

In your lab notebook set up a data and results table in the format shown in Part 1b of the **Report Form**. Record data in the table as you proceed.

Using a volumetric pipet for the **0.00200 M** $\text{Fe}(\text{NO}_3)_3$, and graduated pipets for the KSCN and HNO_3 , prepare the following solutions in clean, dry, labeled test tubes that will easily hold 10 mL of solution and allow mixing.

Equilibrium Solution #	0.00200 M $\text{Fe}(\text{NO}_3)_3$ (in 0.1 M HNO_3), mL	2.00×10^{-3} M KSCN (in 0.1 M HNO_3), mL	0.1 M HNO_3
E1 (blank)	5.00	0.00	5.00
E2	5.00	1.00	4.00
E3	5.00	2.00	3.00
E4	5.00	3.00	2.00
E5	5.00	4.00	1.00

Mix each solution thoroughly for 1 to 2 minutes. **Make up all five solutions before measuring absorbances**. As in Part 1, zero the spectrophotometer, and then determine and record the absorbances of solutions E2 through E4 against E1 as a blank.

b) Calculating the Concentration of FeSCN^{2+} in Equilibrium Solutions

After lab, working in your lab notebook, set up a table for your calculated results, using the table format shown in the **Report Form**. Use the calibration equation obtained in Part 1c to calculate $[\text{FeSCN}^{2+}]$ in each of the equilibrium solutions E2 through E4.

Lab Procedure

c) Calculating K_C

Working in your lab notebook, calculate the initial concentrations of Fe^{3+} and SCN^- in solutions E2 through E4. From the measured concentrations of FeSCN^{2+} in each equilibrium solution (calculated in part 1b), calculate the **final** concentrations of Fe^{3+} and SCN^- (use an ICE table). Enter all results in the table.

For each equilibrium solution, compute K_C . Enter results in the table of your lab notebook. Determine the mean and standard deviation for your four determinations of K_C . In your notebook, comment on the level of agreement among your values.

Complete the **Report Form** for this experiment.