Experiment 9. An Equilibrium Constant

Experimental Procedure Lab 402

Overview

One set of solutions having known molar concentrations of $FeNCS^{2+}$ is prepared for a calibration curve, a plot of absorbance versus concentration. A second set of equilibrium solutions is prepared and mixed to determine the respective equilibrium molar concentrations of $FeNCS^{2+}$. By carefully measuring the initial amounts of reactants placed in the systems and the absorbance, the mass action expression at equilibrium can be solved; this equals K_c.

Part A.
A Set of Standard Solutions to Establish a Calibration Curve

1. Prepare a set of the standard solutions

1) Prepare the solutions in Table 34.1. Pipet 0, 1, 2, 3, 4 and 5 mL of 0.001 M NaSCN into separate, labeled, and clean 50-mL mass cylinder. Pipet 10.0 mL of 0.2 M $Fe(NO_3)_3$ into each flask and quantitatively dilute to 25 mL with 0.1 M HNO_3. Stir or agitate each solution thoroughly to ensure that equilibrium is established. Record on the Report Sheet the exact molar concentrations of the $Fe(NO_3)_3$ and NaSCN reagent solutions, i.e, the correct number of significant figures.

Table 34.1 Composition of the Set of Standard $FeNCS^{2+}$ Solutions for Preparing the Calibration Curve

Standard Solution	0.2 M $Fe(NO_3)_3$ (in 0.1 M HNO_3)	0.001 M NaSCN (in 0.1 M HNO_3)	0.1 M HNO_3
Blank	10.0 mL	0 mL	Dilute to 25 mL
1	10.0 mL	1 mL	Dilute to 25 mL
2	10.0 mL	2 mL	Dilute to 25 mL
3	10.0 mL	3 mL	Dilute to 25 mL
4	10.0 mL	4 mL	Dilute to 25 mL
5	10.0 mL	5 mL	Dilute to 25 mL

2. Prepare the blank solution

After the spectrophotometer has been turned on for 10 minutes and the wavelength scale has been set at ### nm, rinse a cuvet with several portions of the blank solutions. Dry the outside of the cuvet with a clean Kimwipe, removing water and fingerprints. Handle the lip of the cuvet thereafter. If a cuvet has two clear and two cloudy sides, be sure light passes through the clear sides and handle the cuvet on the cloudy sides.

3. Calibrate the spectrophotometer

Place the cuvet, three-fourths filled with the blank solution, into the sample compartment, align the mark on the cuvet with that on the sample holder, and close the cover. Set the meter on the spectrophotometer to read zero absorbance. Remove the cuvet.

4. Record the absorbance of the standard solutions

Empty the cuvet and rinse it thoroughly with several small portions of ***Solution 1****. Fill it approximately three-fourths full. Again, carefully dry the outside of the cuvet with a clean Kimwipe. Remember, handle only the lip of the cuvet. Place the cuvet into the sample compartment and align the cuvet and sample holder marks; read the absorbance and record. Repeat for Solutions 2, 3, 4, and 5.*

Share the set of standard solutions with other groups in the laboratory.

5. Graph the data.

Plot absorbance, A (ordinate), versus [$FeNCS^{2+}$] (abscissa) for the six solutions by using appropriate software. Draw the best straight line through the six points to establish the calibration curve.

Part B.
Absorbance for the Set of Test Solutions

1. Prepare the test solutions.

In clean 150-mm test tubes (or 10-mL volumetric flasks) prepare the test solutions in Table 34.2. Use pipets for the volumetric measurements. Be careful not to mix pipets to avoid contamination of the reagents prior to the preparation. Also note that the molar concentration of $Fe(NO_3)_3$ for this set of solutions is 0.002 M, not the 0.2 M solution used in PART A and the molar concentration of NaSCN in 0.002, not 0.001 M.

Record the exact molar concentration of $Fe(NO_3)_3$ and NaSCN reagent solutions on the Report Sheet, PART B, i.e, the correct number of significant figures.

Once the test solutions are prepared, proceed smoothly and methodically through PART B.3.

Table 34.2 Composition of the Set of Equilibrium Test Solutions for the Determination of K_c.

Test Solution	0.002 M $Fe(NO_3)_3$ (in 0.1 M HNO_3)	0.002 M NaSCN (in 0.1 M HNO_3)	0.1 M HNO_3
6	5 mL	1 mL	4 mL
7	5 mL	1 mL	3 mL
8	5 mL	1 mL	2 mL
9	5 mL	1 mL	1 mL
10	5 mL	1 mL	-

2. Recalibrate the spectrophotometer

Use the blank solution for PART A to check the calibration of the spectrophotometer.

3. Recalibrate the spectrophotometer

Stir or agitate each test solution until equilibrium is reached (approximately 30 seconds). Rinse the cuvet thoroughly with several portions of the test solution and fill it three-fourths full. Clean and dry the outside of the cuvet. Be cautious in handling the cuvets. Record the absorbance of each test solutions as was done PART A.4.

4. Use data to determine equilibrium concentrations.

From the calibration curve prepared in PART A.5, use the recorded absorbance value for each test solution to determine the equilibrium molar concentration of $FeNCS^{2+}$ and record on the Report Sheet, lines C.1.

Part C.
Calculation of K_c

1. Data analysis

Complete the calculations as outlined on the Prelaboratory Assignment, question 6, the Report Sheet and described in the Introduction. Complete an entire K_c calculation for Test Solution 6 before attempting the calculations for the remaining solutions.

The equilibrium constant will vary from solution to solution and from chemist to chemist in this experiment, depending on chemical technique and the accumulation and interpretation of the data. Consequently, it is beneficial to work through your own calculations with other colleagues.

Disposal:

Dispose of all waste thiocyanatoiron(III) ion solutions form Part A and B in the Waste Salts container.

CLEANUP:

Rinse the volumetric flasks, the pipets, and the cuvets twice with tap water and twice with deionized water. Discard each time in the sink.