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## The Determination of $K_{eq}$ for $FeSCN^{2+}$ AP Chemistry Laboratory #17

### Introduction

For any reversible chemical reaction at equilibrium, the concentrations of all reactants and products are constant or stable. There is no further net change in the amounts of reactants and products unless the reaction mixture is disturbed in some way. The equilibrium constant provides a mathematical description of the position of equilibrium for any reversible chemical reaction. What is the equilibrium constant and how can it be determined?

### Concepts

- Chemical equilibrium
- Equilibrium constant
- Complex-ion reaction
- Colorimetry

### Background

Any reversible reaction eventually reaches a position of *chemical equilibrium*. In some cases, equilibrium favors products and it appears that the reaction proceeds essentially to completion. The amount of reactants remaining under these conditions is very small. In other cases, equilibrium favors reactants and it appears that the reaction occurs only to a slight extent. Under these conditions, the amount of products present at equilibrium is very small.

These ideas can be expressed mathematically in the form of the equilibrium constant. Consider the following general equation for a reversible chemical reaction:



The *equilibrium constant*  $K_{eq}$  for this general reaction is given by Equation 2, where the square brackets refer to the molar concentrations of the reactants and products at equilibrium.

$$K_{eq} = \frac{[C]^c[D]^d}{[A]^a[B]^b} \quad \text{Equation 2}$$

The equilibrium constant gets its name from the fact that for any reversible chemical reaction, the value of  $K_{eq}$  is a constant at a particular temperature. The concentrations of reactants and products at equilibrium vary, depending on the initial amounts of materials present. The special ratio of reactants and products described by  $K_{eq}$  is always the same, however, as long as the system has reached equilibrium and the temperature does not change. The value of  $K_{eq}$  can be calculated if the concentrations of reactants and products at equilibrium are known.

The reversible chemical reaction of iron(III) ions ( $Fe^{3+}$ ) with thiocyanate ions ( $SCN^-$ ) provides a convenient example for determining the equilibrium constant of a reaction. As shown in Equation 3,  $Fe^{3+}$  and  $SCN^-$  ions combine to form a special type of combined or "complex" ion having the formula  $FeSCN^{2+}$ .



The equilibrium constant expression for this reaction is given in Equation 4.

$$K_{\text{eq}} = \frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^{-}]} \quad \text{Equation 4}$$

The value of  $K_{\text{eq}}$  can be determined experimentally by mixing known concentrations of  $\text{Fe}^{3+}$  and  $\text{SCN}^{-}$  ions and measuring the concentration of  $\text{FeSCN}^{2+}$  ions at equilibrium. As noted in Equation 3, the reactant ions are pale yellow and colorless, respectively, while the product ions are blood-red. The concentration of  $\text{FeSCN}^{2+}$  complex ions at equilibrium is proportional to the intensity of the red color.

A special sensor or instrument called a *colorimeter* can be used to measure the absorbance of light by the red ions. The more intense the red color, the greater the absorbance. The wavelength of light absorbed by the red ions is about 450 nm. None of the other ions present in solution absorb light at this wavelength. As long as the same size container is used to measure the absorbance of each solution, the absorbance is directly proportional to the concentration of  $\text{FeSCN}^{2+}$  ions.

### Experiment Overview

The purpose of this experiment is to calculate the equilibrium constant for the reaction of iron(III) ions with thiocyanate ions. The reaction is tested under different conditions to determine if the equilibrium constant always has the same numerical value. There are two parts to the experiment.

In Part A, a series of reference solutions and test solutions are prepared. The reference solutions are prepared by mixing a large excess of  $\text{Fe}^{3+}$  ions with known amounts of  $\text{SCN}^{-}$  ions. According to LeChâtelier's Principle, the large excess of iron(III) ions should effectively convert all of the thiocyanate ions to the blood-red  $\text{FeSCN}^{2+}$  complex ions. The concentration of  $\text{FeSCN}^{2+}$  complex ions in the reference solutions is essentially equal to the initial concentration of  $\text{SCN}^{-}$  ions. The test solutions are prepared by mixing a constant amount of  $\text{Fe}^{3+}$  ions with different amounts of  $\text{SCN}^{-}$  ions. These solutions contain unknown concentrations of  $\text{FeSCN}^{2+}$  ions at equilibrium.

In Part B, the absorbances of both the reference solutions and the test solutions are measured by colorimetry. A calibration curve is constructed from the absorption values of the reference solutions. The unknown concentrations of  $\text{FeSCN}^{2+}$  in the test solutions are calculated by comparing their absorbance readings to the absorbance values of the calibration curve.

### Pre-Lab Questions (Show all work on a separate sheet of paper.)

1. "The equilibrium concentration of  $\text{FeSCN}^{2+}$  ions in each reference solution is essentially equal to the concentration of  $\text{SCN}^{-}$  ions in solution before any reaction occurs." Use LeChâtelier's Principle to explain why this statement is true.

2. The five reference solutions in Part A are prepared by mixing the 0.200 M  $Fe(NO_3)_3$  solution and the 0.00020 M KSCN solution in the amounts listed in the following table.

Standard	Volume of 0.200 M $Fe(NO_3)_3$ Solution	Volume of 0.0002 M KSCN Solution
Reference solution #1	8.0 mL	2.0 mL
Reference solution #2	7.0 mL	3.0 mL
Reference solution #3	6.0 mL	4.0 mL
Reference solution #4	5.0 mL	5.0 mL
Reference solution #5	4.0 mL	6.0 mL

The concentration of  $Fe^{3+}$  ions in the first reference solution ( $M_2$ ) before any reaction occurs can be calculated using the so-called “dilution equation,” as shown below.

$$M_1V_1 = M_2V_2 \quad \text{Dilution Equation}$$

$M_1$  = concentration of solution before mixing = 0.200 M  $Fe(NO_3)_3$

$V_1$  = volume of solution before mixing = 8.0 mL

$V_2$  = final volume of reference solution after mixing = 8.0 + 2.0 mL = 10.0 mL

$$M_2 = \frac{M_1V_1}{V_2} = \frac{(0.200 \text{ M})(8.0 \text{ mL})}{(10.0 \text{ mL})} = 0.16 \text{ M}$$

Use the dilution equation to calculate the concentration of  $SCN^-$  ions in the five reference solutions before any reaction occurs. Enter these values in the Reference Solutions Data Table as  $[FeSCN^{2+}]$ .

3. The following table summarizes the volumes of  $Fe^{3+}$  and  $SCN^-$  stock solutions that will be mixed together to prepare the test solutions in Part A. Use the dilution equation to calculate the concentrations of  $Fe^{3+}$  and  $SCN^-$  ions in each test solution before any reaction occurs. Enter the results of these calculations in scientific notation in the Test Solutions Data Table. *Hint:* The final volume ( $V_2$ ) of each test solution is 10.0 mL.

Sample	Volume of 0.0020 M $Fe(NO_3)_3$ Solution	Volume of 0.0020 M KSCN Solution	Volume of Distilled Water Added
Test solution #6	5.0 mL	1.0 mL	4.0 mL
Test solution #7	5.0 mL	2.0 mL	3.0 mL
Test solution #8	5.0 mL	3.0 mL	2.0 mL
Test solution #9	5.0 mL	4.0 mL	1.0 mL
Test solution #10	5.0 mL	5.0 mL	0 mL

## Materials

Iron(III) nitrate, $Fe(NO_3)_3$ , 0.200 M, 30 mL <sup>†</sup>	Beakers or large test tubes, 50-mL, 10
Iron(III) nitrate, $Fe(NO_3)_3$ , 0.0020 M, 25 mL <sup>†</sup>	Cuvettes with lids, 6
Potassium thiocyanate, KSCN, 0.0020 M, 15 mL	Labeling or marking pen
Potassium thiocyanate, KSCN, 0.0002 M, 20 mL	Pipets, serological-type, 5- or 10-mL, 5
Water, distilled or deionized	Pipet bulb or pipet filler
Colorimeter sensor or spectrophotometer	Stirring rod
Computer interface system (LabPro), 15*	Tissues or lens paper, lint-free
Computer or calculator for data collection, 15*	Thermometer
Data collection software (LoggerPro)*	Wash bottle

\*Not required if spectrophotometer is used.

<sup>†</sup>Contains 1 M nitric acid as the solvent.

## Safety Precautions

*Iron(III) nitrate solution contains 1 M nitric acid and is a corrosive liquid; it will stain skin and clothing. Notify the teacher and clean up all spills immediately. Potassium thiocyanate is toxic by ingestion; it can generate poisonous hydrogen cyanide gas if heated strongly. Avoid contact of all chemicals with eyes and skin. Wear chemical splash goggles and chemical-resistant gloves and apron. Wash hands thoroughly with soap and water before leaving the laboratory.*

## Procedure

### Part A. Preparing the Solutions

1. Obtain ten 50-mL beakers or large test tubes.
2. Prepare the five reference solution test tubes or beakers listed in the table below. Use a separate pipet to transfer the appropriate volumes of each reagent. Mix each solution using a stirring rod. Rinse the stirring rod and dry it between solutions. Label the test tubes or beakers with the corresponding reference solution number.

Standard	Volume of 0.200 M $Fe(NO_3)_3$ Solution	Volume of 0.00020 M KSCN Solution
Reference solution #1	8.0 mL	2.0 mL
Reference solution #2	7.0 mL	3.0 mL
Reference solution #3	6.0 mL	4.0 mL
Reference solution #4	5.0 mL	5.0 mL
Reference solution #5	4.0 mL	6.0 mL

- Using a separate pipet for each reagent to be added, combine the following volumes of reagents to prepare the test solutions. *Note:* Label the tubes with the corresponding solution numbers 6 through 10. Read the reagent labels carefully before use!

Sample	Reagents		
	0.0020 M $Fe(NO_3)_3$	0.0020 M KSCN	Distilled Water
Test solution #6	5.0 mL	1.0 mL	4.0 mL
Test solution #7	5.0 mL	2.0 mL	3.0 mL
Test solution #8	5.0 mL	3.0 mL	2.0 mL
Test solution #9	5.0 mL	4.0 mL	1.0 mL
Test solution #10	5.0 mL	5.0 mL	0 mL

- Mix each solution using a stirring rod. Rinse the stirring rod and dry it between solutions.
- Measure the temperature of one of the solutions and record it in the Test Solutions Data Table. This is assumed to be the equilibrium temperature for all of the solutions.

### Part B. Colorimetry Measurements

- Follow the procedure for your colorimetric measurements of the solution as directed by the instructor. Generally, spectrophotometers are used as follows: Turn the instrument on and allow it to warm up for 15 minutes. Set the wavelength at 450 nm. With no light passing through the instrument to the phototube, set the percent transmittance to zero with the “zero” control. Handle cuvetts at the top so no fingerprints are in the light path. Polish cuvetts with a tissue. Place a cuvette which is about 2/3 full of distilled water into the sample holder and set the percent transmittance to 100% with the appropriate control (not the zero control). Fill a cuvette about 2/3 full of a test solution, place it in the spectrophotometer and read the absorbance. Consult the instrument manual for details on its use.
- Measure the absorbance of each of the reference solutions at 450 nm, using distilled water as the zero absorbance reference in the spectrophotometer. If absorbance is difficult to measure precisely on the meter because it is in the high range where the numbers are close together, measure percent transmittance and calculate the absorbance for each solution. Absorbance =  $-\log T$ , where  $T$  is transmittance expressed as a decimal. Record the absorbance value for each reference solution used in the Reference Solutions Data Table.
- Repeat step 2 for each of the test solutions. Record the absorbances in the Test Solution Data Table.
- Dispose of the contents of the cuvetts and of the remaining test solutions as directed by your instructor. Follow your instructor’s directions for rinsing and drying the cuvetts.

Name \_\_\_\_\_

**Data Tables****Reference Solutions**

Temperature		
Sample	$[FeSCN^{2+}]$	Absorbance
Reference solution #1		
Reference solution #2		
Reference solution #3		
Reference solution #4		
Reference solution #5		

**Test Solutions**

Temperature			
Sample	$[Fe^{3+}]^*$	$[SCN^-]^*$	Absorbance
Test solution #6			
Test solution #7			
Test solution #8			
Test solution #9			
Test solution #10			

\*These are the concentrations of ions in solution immediately after mixing and before any reaction has occurred. See the *Pre-Lab Questions* for calculations.

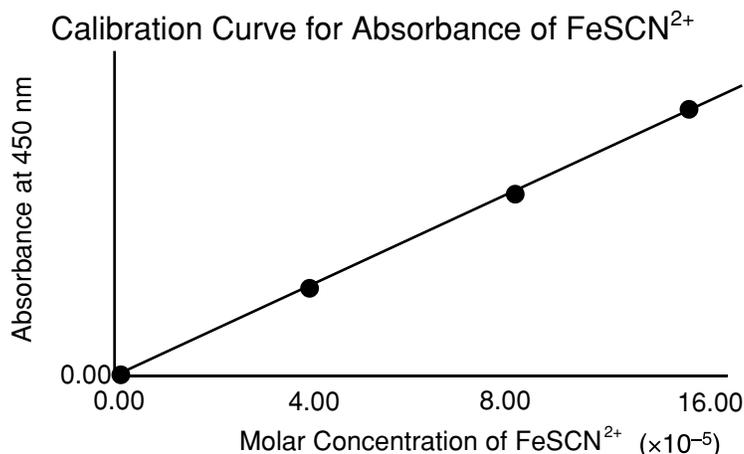
**Results Table**

Sample	$[FeSCN^{2+}]_{eq}$	$[Fe^{3+}]_{eq}$	$[SCN^-]_{eq}$	$K_{eq}$
Test Solution #6				
Test Solution #7				
Test Solution #8				
Test Solution #9				
Test Solution #10				
Average value				
Average deviation				

## Post-Lab Calculations and Analysis

(Use a separate sheet of paper to answer the following questions.)

1. Plot molar concentration of  $FeSCN^{2+}$  versus absorbance as shown in Figure 1, and draw the best-fitting straight line through the data points. Include the origin (zero absorbance for zero concentration) as a valid point.



**Figure 1.** Sample Graph of Absorbance versus Concentration of  $FeSCN^{2+}$

2. The unknown concentration of  $FeSCN^{2+}$  ions in each test solution can be determined from the graph. Find the absorbance value of the test solution, read across to the best-fit, straight-line curve, and then down to the x-axis to find the concentration.
3. Record the  $FeSCN^{2+}$  concentration for each test solution in the Results Table.
4. Calculate the equilibrium concentration of  $Fe^{3+}$  ions in each test solution #6–10: subtract the equilibrium concentration of  $FeSCN^{2+}$  ions from the initial concentration of  $Fe^{3+}$  ions (see the Test Solutions Data Table). Enter the results in the Results Table.

$$[Fe^{3+}]_{eq} = [Fe^{3+}]_{initial} - [FeSCN^{2+}]_{eq}$$

5. Calculate the equilibrium concentration of  $SCN^{-}$  ions in each test solution #6–10: subtract the equilibrium concentration of  $FeSCN^{2+}$  ions from the initial concentration of  $SCN^{-}$  ions (see the Test Solutions Data Table). Enter the results in the Results Table.

$$[SCN^{-}]_{eq} = [SCN^{-}]_{initial} - [FeSCN^{2+}]_{eq}$$

6. Use Equation 4 in the *Background* section to calculate the value of the equilibrium constant  $K_{eq}$  for each test solution #6–10. Enter the results in the Results Table.
7. Calculate the *mean* (average value) of the equilibrium constant for the five test solutions.
8. Calculate the *average deviation* for  $K_{eq}$ : Find the absolute value of the difference between each individual value of the equilibrium constant and the mean. The average of these differences for solutions #6–10 is equal to the average deviation.
9. The average deviation describes the precision of the results. Does the precision indicate that the equilibrium constant is indeed a “constant” for this reaction? Explain.
10. Describe the possible sources of error in this experiment and their likely effect on the results.