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Experiment #7 – Determination of the Equilibrium Constant for the Formation of $FeSCN^{+2}$

When chemical substances react, the reaction typically does not go to completion. Rather, the system goes to some intermediate state in which both the reactants and products have concentrations which do not change with time at a particular temperature. Such a system is said to be at chemical equilibrium. This condition is expressed in the equilibrium constant, K_c, for the reaction.

In this experiment, we will study the equilibrium properties of the complex ion formation reaction between the iron(III) ion and the thiocyanate ion:

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

When solutions containing Fe^{3+} ion and thiocyanate ion are mixed, Reaction 1 occurs to some extent, forming the $FeSCN^{2+}$ complex ion, which has an orange color. As a result of the reaction, the equilibrium amounts of Fe^{3+} and SCN^{-} will be less than they would have been if no reaction occurred; for every mole of $FeSCN^{2+}$ that is formed, one mole of Fe^{3+} and one mole of SCN^{-} will react. According to our studies, the equilibrium constant expression, K_c, for Reaction 1 is formulated as follows:

$$\frac{[FeSCN^{2+}]}{[Fe^{3+}][SCN^{-}]} = K_c \qquad (2)$$

The value of K_c in Equation 2 is constant at a given temperature. This means that mixtures containing Fe^{3+} and SCN^{-} will react until Equation 2 is satisfied so that the same value of the K_c will be obtained no matter what initial amounts of Fe^{3+} and SCN^{-} were used. Our purpose in this experiment will be to find K_c for this reaction involving several mixtures made up in different ways as well as to show that K_c indeed has the same value in each of the mixtures. The reaction studied is a particularly good one because K_c is of a convenient magnitude, the color of the $FeSCN^{2+}$ ion makes for an easy analysis of the equilibrium mixture, and the reaction reaches equilibrium quickly.

The mixtures will be prepared by combining solutions containing known concentrations of iron(III) nitrate, $Fe(NO_3)_3$, and potassium thiocyanate, KSCN in water. The color of the $FeSCN^{2+}$ ion formed will allow us to determine its equilibrium concentration. Knowing the initial composition of a mixture and the equilibrium concentration of $FeSCN^{2+}$, we can calculate the equilibrium concentrations of the rest of the pertinent species and then determine K_c.

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To determine the [$FeSCN^{2+}$] in the equilibrium mixtures, we will use a spectrophotometer, which measures the amount of light absorbed by the orange complex at ***447 nm***, the wavelength at which the complex most strongly absorbs. The absorbance, A, of the complex is proportional to its concentration, M, and can be measured directly on the spectrophotometer. Review the Experiment Six introduction on Beer's Law and procedure on how to calibrate and operate the spectrophotometer. Once the absorbance value is recorded, use the value $\boldsymbol{k = 5.00 \times 10^3}$ to determine the concentration (M) of $FeSCN^{2+}$ for each solution using the equation $\boldsymbol{A = k\,M}$.

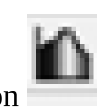
Procedure

Label five regular CLEAN, DRY test tubes 1 to 5 and place them in a test tube rack. Use a buret to deliver the appropriate amount of each reactant into a 10.00 mL volumetric flask. Then add enough D.I. water to bring the solution volume to the 10.00 mL mark. Transfer this mixture to the appropriate labeled test tube. Rinse the volumetric flask with D.I. water and prepare the remaining mixtures in similar fashion. *Prepare mixture 5 by adding the $Fe(NO_3)_3$ and KSCN directly to the test tube; do not use the volumetric flask.* The reagents to be added for each mixture are summarized:

	1	2	3	4	5
Volume $Fe(NO_3)_3$ solution (mL)	5.00	5.00	5.00	5.00	5.00
Volume KSCN solution (mL)	1.00	2.00	3.00	4.00	5.00
H_2O (mL)	Enough to bring total volume to 10.00 mL				

Mix each solution thoroughly with a glass stirring rod and allow them to reach equilibrium for at least 5 minutes. Be sure to DRY THE STIRRING ROD AFTER MIXING EACH SOLUTION!

Instructions for Using Spectrovis with PC.

a) Plug the USB cable from the Spectrovis into the PC.
b) Open Logger Pro on the computer.
c) Under Experiment choose Calibrate then Spectrometer 1.
d) Allow the 90 s warmup.
e) Fill the cuvette (up to about ¾) with the blank solution (blank is just water for this experiment). Insert the cuvette into the Spectrovis. Make sure the clear sides are the ones in the light path.
f) Select Finish Calibration.
g) When OK becomes available, select it.
h) The machine is now calibrated and there is no need to re-calibrate unless you exit out of Logger Pro.
i) Click the Wave or Configure Spectrometer button .
j) Choose the Absorbance vs Concentration option.
k) ON the bottom drop down menu, change from single 10 nm into individual wavelengths.

l) Choose the wavelength closest to λ_{max} for the orange complex (447 nm).
m) Click OK.
n) If one or two pop-up window/s appear/s, choose Erase and Continue AND/OR "No" on the next pop-up window.
o) Click collect (green button).
p) Fill (up to about ¾) your cuvette with one of your five mixtures and insert the cuvette into the Spectrovis.
q) When the absorbance reading stabilize (usually takes a second), click KEEP.
r) On the pop-up window, enter the solution number (e.g. 1, 2, 3 etc..).
s) Discard the solution in the cuvette.
t) Repeat steps p through s for your other solutions.
u) When you have measured ***ALL*** of your standard solutions, Click STOP to end data collection. ***DO NOT EXIT OUT OF LOGGER PRO SO THE NEXT USER WILL NOT HAVE TO CALIBRATE THE MACHINE.***
v) Write down the absorbance values for each of your five solutions on the data table.

**** If you are the second or third user of the Spectrovis, start from step o above (Click the collect (green button)).***

Calculate K_c assuming the reaction: $Fe^{3+}(aq) + SCN^{-}(aq) \rightleftharpoons FeSCN^{2+}(aq)$.

Step 1. Calculate the equilibrium concentration of $FeSCN^{2+}$ from the absorbance using $A = k[FeSCN^{2+}]$, where $k = 5.00 \times 10^3$.

Step 2. Find the new initial concentrations of Fe^{3+} and SCN^{f-} in the mixtures in test tubes 1 through 5 by performing a dilution calculation. Enter the values in the first two columns of the table.

Step 3. Enter the experimentally determined value of $[FeSCN^{2+}]$ at equilibrium for each of the mixtures in column 5 of the table.

Step 4. From the concentration of Fe^{3+} and SCN^{-} initially present in each mixture and the equilibrium concentration of $FeSCN^{2+}$, calculate the concentration of Fe^{3+} and SCN^{-} that remain in each mixture at equilibrium. Enter these results in columns 3 and 4 in the table.

Step 5. Calculate K_c for the reaction for each mixture by substituting values for the equilibrium concentrations of Fe^{3+}, SCN^{-}, and $FeSCN^{2+}$ into Eq. 2.

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Pre-Lab Questions: Equilibrium Constant Determination for $FeSCN^{+2}$

1. A student mixes 5.00 mL of 2.00×10^{-3} M $Fe(NO_3)_3$ with 5.00 mL of 2.00×10^{-3} M KSCN. She finds that in the equilibrium mixture, the concentration of $FeSCN^{2+}$ is 1.40×10^{-4} M. Find K_c for $Fe^{3+}(aq) + SCN^{-}(aq) \rightleftharpoons FeSCN^{2+}(aq)$.

Step 1. Calculate the initial, diluted concentrations of the Fe^{3+} and SCN^{-} ions in the total of 10.00 ml solution using $M_1V_1 = M_2V_2$

$[Fe^{3+}]$____________ $[SCN^{-}]$____________

Step 2. Use the initial concentrations of the Fe^{3+} and SCN^{-} ions along with the equilibrium concentration of the $FeSCN^{2+}$ ion and the reaction stoichiometry to determine the equilibrium concentrations of Fe^{3+} and SCN^{-}.

	$[Fe^{3+}]$	+	$[SCN^{-}]$	$\leftrightharpoons$	$[FeSCN^{2+}]$
initial	_________		_________		_________
Δ	_________		_________		_________
equil	_________		_________		_________

Step 3. Solve for the value of K_c for the reaction. (Use Eq. 2 and the results of Step 2.)

K_c = ____________

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Data: Determination of the Equilibrium Constant for the Formation of $FeSCN^{+2}$

Mixture	Vol. of 2.00×10^{-3} M $Fe(NO_3)_3$ (in mL)	Vol. of 2.00×10^{-3} M KSCN (in mL)	Vol. of H_2O (in mL)	Absorbance
1	5.00	1.00	4.00	______
2	5.00	2.00	3.00	______
3	5.00	3.00	2.00	______
4	5.00	4.00	1.00	______
5	5.00	5.00	0.00	______

Mixture	Initial Concentrations AFTER MIXING		Equilibrium Concentrations			
	$[Fe^{+3}]$	$[SCN^-]$	$[Fe^{+3}]$ +	$[SCN^-]$	$\leftrightharpoons$ $[FeSCN^{+2}]$	K_c
1	______	______	______	______	______	______
2	______	______	______	______	______	______
3	______	______	______	______	______	______
4	______	______	______	______	______	______
5	______	______	______	______	______	______

Hint: Set up each mixture as shown in the pre-lab *Reaction-Initial-Change-Equilibrium*

Average K_c ______________

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Post-Lab Questions: Determination of the Equilibrium Constant for the Formation of $FeSCN^{+2}$

1. Are the K_c values on the previous page consistent? If not, suggest a reason for any large differences.

2. In carrying out this analysis, we made the assumption that the reactants were reacting as a 1:1 mole ratio, as given by Equation 1. There is no inherent reason why the reaction might not have been a 1:2 mole ratio:

$$Fe^{3+}(aq) + 2\ SCN^{-}(aq) \rightleftharpoons Fe(SCN)_2^{+}(aq) \qquad (3)$$

   a. Fill in the equilibrium values in the chart below using your experimental data and this new reaction ratio:

Reaction	**$Fe^{+2}(aq)$ +**	**$2\ SCN^{-}(aq)$ $\rightleftharpoons$**	**$Fe(SCN)_2^{+}(aq)$**
test tube 1 mixture at equilibrium			
test tube 5 mixture at equilibrium			

   b. Calculate the value of K_c using the data from the test tube 1 mixture, assuming that the reaction is actually the one shown in equation 3.

   c. Calculate the value of K_c using the data from the test tube 5 mixture, assuming that the reaction is actually the one shown in equation 3.

   d. Compare the K_c values that you calculated in parts a and b above. Are they consistent? Do you think Reaction 3 is occurring?