Determining An Equilibrium Constant Using Spectrophotometry and Beer's Law

Objectives:

- 1.) To determine the equilibrium constant for the reaction of iron (III) and thiocyanate to form the thiocyanatoiron(III) complex ion using spectrophotometric data.
- 2.) To determine the concentration of an unknown by evaluating the relationship between color intensity and concentration.

Background Information:

One of the fundamental problems in chemistry is how to determine the extent of a reaction. While not every chemical reaction goes to completion, they usually approach an equilibrium state. When the system reaches equilibrium, the concentrations of the reactants and products no longer change over time. This does not mean that the reaction has ceased. In fact, the reaction continues to progress forward, as well as backward. It is said to be in a dynamic state of equilibrium where the rate of the products being formed from the reactants is exactly the same as the rate of the products being decomposed to form the reactants.

For the general equilibrium reaction

$$aA + bB \le cC + dD$$
 (Eqn. 1)

When studying the equilibrium of chemical systems, one of the most important quantities to determine is the **equilibrium constant**, K_{eq} . At equilibrium at a given temperature, the mass action expression is a constant, known as the equilibrium constant, K_{eq} . The equilibrium expression for the reaction in Equation 1 is given as:

$$K_{eq} = [\underline{C}]^{c} [\underline{D}]^{d}$$

$$[A]^{a} [B]^{b}$$
(Eqn. 2)

The value of the equilibrium constant may be determined from experimental data if the concentrations of both the reactants and the products are known. Additionally, all equilibrium concentrations can be calculated if a single equilibrium concentration is known along with all other "initial" concentrations.

It may be recalled that in spectrophotometric studies, the Beer-Lambert law, or Beer's Law, can be used to determine the concentration of highly colored species. Mathematically, Beer's Law can be stated as:

$$A = abc (Eqn. 3)$$

where "a" is the molar absorptivity, "b" is the pathlength, "c" is the concentration, and "A" is absorbance. **Molar absorptivity**, a, is a proportionality constant that has a specific value for each absorbing species at a given wavelength. The **pathlength**, b, is the distance across the solution in centimeters and is dependent upon the size of the cuvette. In this case, the pathlength will be kept constant at 1.00 cm. The **concentration**, c, of the absorbing species is in moles of solute per liter of solution.

Absorbance is mathematically defined as:

$$A = \log(I_0/I)$$
 (Eqn. 4)

where I_o is the initial intensity of the beam prior to going through the solution and I is the intensity of the beam after it has been transmitted through the solution.

The measurements on the spectrophotometers will be taken in units of Percent Transmittance (%T) with values varying from 0-100. Because of the linear nature of the %T scale and the limited range of possible values, this scale is considered more precise than the Absorbance scale. In this experiment, all readings will be taken in units of %T and the Absorbance will be calculated using the following equation:

$$A = \log [100 / (\%T)]$$
 (Eqn. 5)

When combined iron (III) and thiocyanate ions form "blood red" complexes. So in this experiment, spectrophotometric methods will be used to determine the concentration of the iron (III) cyanato complex, [FeSCN $^{2+}$]. This however is difficult because the thiocyanate ion, SCN $^{-}$, can react with the ferric ion, Fe $^{3+}$, in acidic solutions to form a series of thiocyanato- complexes: Fe(SCN) $^{2+}$, Fe(SCN) $^{2+}$, Fe(SCN) $^{3-}$, and Fe(SCN) $^{4-}$.

The iron (III) ion also introduces a complication because of its reaction with water to form iron hydroxide, which is insoluble in water:

$$Fe^{3+}_{(aq)} + 3 H_2O_{(l)} \le Fe(OH)_{3(s)} + 3 H^+_{(aq)}$$
 (Eqn. 6)

To avoid precipitation of iron (III) hydroxide, you will include excess nitric acid (HNO₃) in all solutions, to shift this equilibrium far to the left. Because neither hydrogen ions nor nitrate ions are components of the iron (III) thiocyanate equilibrium, nitric acid does not affect the equilibrium position of the reaction that produces FeSCN ²⁺.

$$Fe^{3+} <=> FeSCN^{2+} = Fe(SCN)_2^+ \dots etc$$
 (Eqn. 7)

When high concentrations of thiocyanate are present, the higher order complexes are predominant in the solution. However, if the molarity of thiocyanate is very low, the only complex formed in any appreciable amount is the monothiocyanatoiron (III) ion.

$$Fe^{3+} + SCN^{-} = FeSCN^{2+}$$
 (Eqn. 8)

With the equilibrium constant:

$$K_{eq} = \underline{[FeSCN^{2+}]}$$

$$[Fe^{3+}][SCN^{-}]$$
(Eqn. 9)

To evaluate the equilibrium constant for this reaction, one must first determine the concentrations of the three ions. There are several different ways to find out these concentrations. An easy way to make these determinations is to use spectrophotometric methods. The thiocyanate ion is colorless. The ferric ion is yellowish. The iron (III) cyanato complex, on the other hand, is a deep red.

The thiocyanatoiron (III) complex absorbs radiation at 447 nm. So at this wavelength, Beer's Law can be rewritten as:

$$A = ab[FeSCN^{2+}]$$
 (Eqn. 10)

This equation can be rearranged to form the equation:

$$[FeSCN^{2+}] = A / ab$$
 (Eqn. 11)

Then A / ab can be substituted for [FeSCN²⁺] into the numerator of Equation.

$$K_{eq} = \frac{(A/ab)}{[Fe^{3+}][SCN^{-}]}$$
 (Eqn. 12)

For the denominator, the calculations get a little more complex. In order to determine the concentration of Fe^{3+} , one must first assume that all of the iron in the system is either in the form of either Fe^{3+} or $FeSCN^{2+}$. By letting $[Fe^*]$ represent the iron initially added to the system, then one can write:

$$[Fe^*] = [Fe^{3+}] + [FeSCN^{2+}]$$
 (Eqn. 13)

This equation can be rearranged to form the equation:

$$[Fe^{3+}] = [Fe^*] - [FeSCN^{2+}]$$
 (Eqn. 14)

Then from Eqn. 10, A / ab can be substituted for [FeSCN²⁺]

$$[Fe^{3+}] = [Fe^*] - (A/ab)$$
 (Eqn. 15)

Likewise, the concentration of the thiocyanate ion, SCN⁻, can be determined. By letting [SCN*] represent the thiocyanate initially added to the system, then one can write:

$$[SCN^*] = [SCN^-] + [FeSCN^{2+}]$$
 (Eqn. 16)

Then from Eqn. 10, A / ab can be substituted for [FeSCN²⁺]

$$[SCN^{-}] = [SCN^{*}] - (A/ab)$$
 (Eqn. 17)

By substituting Equations 10, 14 & 16 into Equation 8:

$$K_{eq} = \frac{(A / ab)}{[[Fe^*] - (A / ab)][[SCN^*] - (A / ab)]}$$
 (Eqn. 18)

Equation 17 contains two unknowns, K and (A / ab). As shown by Frank Oswalt, the following equation can be derived from Equation 17:

$$\frac{A}{[Fe^*][SCN^*]} = \underline{A([Fe^*] + [SCN^*]) K} + abK$$
 (Eqn. 19)

Equation 18 closely resembles the equation for a line: y = mx + b. Then one can plot y vs. x and determine the slope, where:

$$y = \underline{A} \qquad x = \underline{A ([Fe^*] + [SCN^*])}$$
 (Eqn. 20)

$$[Fe^*] [SCN^*]$$

The slope of the resulting straight line is K_{eq} .

Example:

Some students were doing an experiment using copper (II) and thiocyanate ions to form the thiocyanatocopper (II) ion. The thiocyanatocopper(II) ion can be used to demonstrate the calculations. There is a similar equilibrium among Cu^{2^+} , SCN^- and $CuSCN^+$. The equation for the formation of the $CuSCN^+$ is

$$Cu^{2+}_{(aq)} + SCN^{-}_{(aq)} = CuSCN^{+}_{(aq)}$$
 (Eqn. 21)

This reaction can also be studied spectrophotometrically. The thiocyanatocopper(II) ion absorbs radiation at 380 nm whereas the copper (II) and thiocyanate ions do not.

To determine the concentration plot the following values:

$$y = \underline{A} \qquad x = \underline{A ([Cu^*] + [SCN^*])}$$
 (Eqn. 22)
$$[Cu^*] [SCN^*]$$

The slope of the resulting straight line is K_{eq} .

Student A pipetted exactly 10.0 ml of $5.21 \times 10^{-2} \text{ M KSCN}$ solution into a volumetric flask. He then added exactly 25.0 ml of 2.0 M nitric acid. He then diluted the solution with distilled H_2O to the 100 ml mark on the flask.

This KSCN-HNO $_3$ solution was transferred to a 250 ml beaker. Student B pipetted exactly 1.00 ml of a 2.515 x 10^{-1} M Cu(NO $_3$) $_2$ in 0.5 M HNO $_3$ into the beaker. After the solution reached equilibrium, Student B poured some of the sample into a cuvette and measured the %T at 380 nm.

The sample was returned to the beaker. Another 1.00 ml of a $2.515 \times 10^{-1} \, M \, Cu(NO_3)_2$ in 0.5 M HNO₃ was added to the beaker. After the solution had again reached equilibrium, Student A poured some of the sample into a cuvette and measured its %T at 380 nm.

This procedure was repeated until 10.0 ml of the copper solution had been added to the KSCN-HNO₃ solution. The following data were obtained:

	Vol. M Cu(NO ₃) ₂ in		
Soln #	HNO ₃ (ml)	%T	A
1	1.00	74.7	0.127
2	2.00	60.1	0.221
3	3.00	50.7	0.295
4	4.00	44.0	0.357
5	5.00	39.6	0.402
6	6.00	35.6	0.449
7	7.00	33.1	0.480
8	8.00	31.1	0.507
9	9.00	29.5	0.530
10	10.00	28.2	0.550

Table 1: The solution mixtures, their transmittances and calculated absorbances.

The equilibrium constant can be determined using the following calculations:

1. The Absorbances were calculated using the following equation:

$$A = log (100 / \%T)$$

For the fist value %T = 74.7

$$A = log (100 / 74.7) = 0.127$$

2. The Molarity of the diluted KSCN solution in the 250 ml beaker before any Cu^{2+} was added:

(10.0 ml) (5.21 x
$$10^{-2}$$
 mmole/ml) = 5.21 x 10^{-1} mmole/ml
5.21 x 10^{-1} mmole/ml / 100.0 ml = 5.21 x 10^{-3} mmole/ml
= 5.21 x 10^{-3} mole/L

3. The calculation of the concentration of copper and thiocyanate ions initially added, [Cu*] and [SCN*] respectively:

For the total volume:

$$V_{total} = 100.00 \text{ ml} + 1.00 = 101.00 \text{ ml}$$

mmole of Cu* = (1.00 ml) (2.515 x 10⁻¹ mmole/ml)
= 2.515 x 10⁻¹ mmole

Then

[Cu*] =
$$(2.515 \times 10^{-1} \text{ mmole/ml}) / (101.00 \text{ ml})$$

= $2.49 \times 10^{-3} \text{ mmole/ml}$
= $2.49 \times 10^{-3} \text{ mole/L}$

Then

[SCN*] =
$$(5.21 \times 10^{-1} \text{ mmole}) / (101.00 \text{ ml})$$

= $5.16 \times 10^{-3} \text{ mmole/ml}$
= $5.16 \times 10^{-3} \text{ mole/L}$

4. Calculation of the total concentration of copper and thiocyanate ions initially added,

$$[Cu^*] + [SCN^*]$$
:

$$[Cu^*] + [SCN^*] = 2.49 \times 10^{-3} \text{ mole/L} + 5.16 \times 10^{-3} \text{ mole/L}$$

= 7.65 x 10⁻³ mole/L

5. Calculation of [Cu*] [SCN*]:

[Cu*] [SCN*] =
$$(2.49 \times 10^{-3} \text{ M}) (5.16 \times 10^{-3} \text{ M})$$

= $1.28 \times 10^{-5} \text{ M}^2$

6. Calculation of A([Cu*] + [SCN*]):

$$A([Cu^*] + [SCN^*]) = (0.127) [(2.49 \times 10^{-3} \text{ M}) + (5.16 \times 10^{-3} \text{ M})]$$

= 9.71 x 10⁻⁴ M²

7. Calculation of A / ([Cu*] [SCN*])

[*Note:* This will be plotted on the y-axis.]

$$y = A / ([Cu^*] [SCN^*]) = (0.127) (1.28 \times 10^{-5} M^2)$$

= 9.92 x 10³ M⁻²

8. Calculation of $A([Cu^*] + [SCN^*]) / ([Cu^*] [SCN^*])$

[*Note:* This will be plotted on the x-axis.]

$$x = A([Cu^*] + [SCN^*]) = 9.71 \times 10^{-4} M^2 / 1.28 \times 10^{-5} M^2$$

 $([Cu^*] [SCN^*]) = 7.59 \times 10^1 M^{-1}$

Calculations 2-8 were repeated for each of the other nine equilibrium mixtures. The following table summarizes the results of these calculations.

Soln #	X	y
1	75.9	9920
2	88.1	8780
3	98.6	7960
4	108	7360
5	115	6770
6	122	6380
7	128	5990
8	132	5640
9	136	5340
10	140	5080

Table 2: The solution mixtures and the calculated values for x & y.

This data was then plotted in Figure 1.

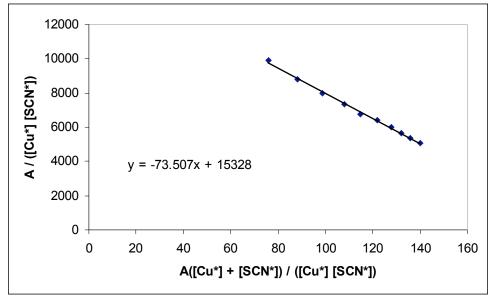


Figure 1: Plot to determine the equilibrium constant for the formation of CuSCN*.

Procedure:

Caution: All of the following chemicals are highly corrosive. Use care in handling the solution used in this experiment. Wash any solution off your skin immediately.

2 M HNO₃ 0.5 M HNO₃ Fe(NO₃)₃-HNO₃ (aq) KSCN-HNO₃ (aq)

Caution: Always wear departmentally approved goggles while doing experiments.

Overview of Procedure:

In Part A of the experiment, the appropriate wavelength to be used in this experiment will be determined. The blank for this experiment will 0.5M HNO₃ instead of distilled water because the Fe(NO₃)₃ was prepared in HNO₃. A known solution of Fe(NO₃)₃ will then be used to determine where the minimum percent transmittance / maximum absorbance occurs.

In Part B, a series of calibration solutions with known concentrations of the iron (III) thiocyanate ion will be prepared. To do this, a large excess of thiocyanate will be added to the Fe ³⁺ to drive the reaction to the left, incorporating all of the ferric ion into FeSCN ²⁺.

In each of these solutions, therefore, the equilibrium concentration of $FeSCN^{2+}$ equals the initial concentration of Fe^{3+} . The equation for the trendline of the Beer's law plot will be used to determine the equilibrium constant K_{eq} for this reaction.

The absorbance of each solution will be determined. A Beer's law plot should be made in Excel (or other graphing program). A linear trendline should be added. The trendline is the equation of the line relating Absorbance to Concentration.

Group A – Determination of Lambda Max for the Iron

- 1. Acquire cuvettes with 0.5M HNO₃ and Fe(SCN)₃ solution from the stockroom.
- 2. Set the spectrophotometer at 400 nm. Zero the spectrophotometer.
- 3. Place the cuvette with the 0.5M HNO₃, the reference solution in the holder and adjust the light control knob to 100%. Remove the cuvette.
- 4. Place the cuvette with the solution in the holder.
- 5. Record the % Transmittance on Datasheet 1.
- 6. Change to the next wavelength and repeat 2-5.
- 7. Calculate the Absorbance for each of the % Transmittance values.
- 8. Record the wavelength that corresponds to the maximum absorbance.

Use this wavelength for Part B.

Group B – Prepare the diluted ferric nitrate solution:

- 1. On Datasheet 1, record the exact molarity of the Fe(NO3)₃ solution and the KSCN solution. Be sure to include all significant digits.
- 2. Condition a 10 ml pipet with the stock solution.
 - a. Acquire ~15 ml of 0.00250 M Fe(NO₃)₃ solution in a 50 ml beaker. (*Note:* The stock solution of 0.00250 M Fe(NO₃)₃ solution was prepared using 0.50 M HNO₃ as the solvent. The solution is highly corrosive. Use caution when working with the solution.)
 - b. Draw ~ 10 ml of Fe(NO₃)₃ from the beaker into the pipet. Fill the pipet to slightly above the 10 ml mark being careful not to overfill the pipet and not to pull the solution into the bulb.
 - c. Remove the bulb and quickly cover the end with your thumb (or forefinger) to create a vacuum.

- d. Gently rock thumb away from the pipet and back to allow air in and practice releasing small amounts of acid into the original 50 ml beaker.
- e. Repeat Steps b-d twice more.
- f. Pour the Fe(NO₃)₃ used for conditioning into the waste container.
- 3. Acquire \sim 15 ml of 0.00250 M Fe(NO₃)₃ solution in the 50 ml beaker.
- 4. Pipet 10.00 ml of Fe(NO₃)₃ solution into a 100 ml volumetric flask.
 - a. Fill the pipet to slightly above the 10 ml mark being careful not to overfill the pipet and not to pull the solution into the bulb.
 - b. Gently rock thumb away from the pipet and back to allow air in and release a small amount of acid until the bottom of the meniscus is exactly on the 10 ml line and then reseal.
 - c. Transfer the pipet tip into a volumetric flask and allow the solution to flow into the flask.
- 5. Acquire \sim 90 ml of 0. 5 M HNO₃ in a dispenser bottle.
- 6. Using a funnel, add \sim 15 ml portions of the HNO₃ to the volumetric slowly.

After adding \sim 15 ml stop and swirl. Then continue with the next addition. When you get close to the fill line, add the HNO₃ dropwise until the bottom of the meniscus is on the line.

7. Once the flask is filled to the 100 ml mark, place the cap on securely.

Invert the flask, shake well, and then revert. Repeat shaking process 10 times.

- 8. Pour the contents of the volumetric flask into a clean dry 250 ml beaker. Label this beaker as "Diluted Fe³⁺".
- 9. Calculate the final iron concentration and record on Datasheet 1.
- 10. Acquire ~25 ml of 0.0025M KSCN in a clean dry 50 ml beaker.
- 11. Pipet 2.00 ml of KSCN into the beaker of "Diluted Fe³⁺".

Stir the mixture with a stirring rod. Be careful to knock any solution off the stirring rod back into the beaker.

- 12. Pour ~8 ml of solution into a cuvette.
- 13. At the appropriate wavelength:
 - a. Zero the spectrophotometer.
 - b. Place the cuvette with the 0.5M HNO₃, the reference solution in the holder and adjust the light control knob to 100%. Remove the cuvette.
 - c. Place the cuvette with the solution in the holder.
 - d. Record the % Transmittance on Datasheet 1.
 - e. Pour the solution out of the cuvette back into the beaker of "Diluted Fe³⁺".
- 14. Pipet another 2.00 ml of KSCN into the beaker of "Diluted Fe³⁺".

Stir the mixture with a stirring rod. Be careful to knock any solution off the stirring rod back into the beaker.

- 15. Pour ~8 ml of solution into a cuvette.
- 16. Place the cuvette with the solution in the holder.
- 17. Record the % Transmittance on Datasheet 1.
- 18. Pour the solution out of the cuvette back into the beaker of "Diluted Fe³⁺".
- 19. Repeat steps 14-18 to obtain the remaining data.
- 20. Dispose of all solutions in the appropriate waste container.
- 21. Clean all of the used glassware and return the appropriate items to the stockroom.

Calculations:

Do the following calculations for each determination and record the results on the appropriate datasheet. (For how to do the calculations, refer to the example of Cu(NO₃)₂.)

- 1. Calculate the molarity of the original diluted Fe(NO₃)₃ solution.
- 2. Calculate the following for each of the 10 equilibrium solutions.
 - a. Convert %T to A.

$$A = log (100 / \%T)$$

b. Calculate [FE*] and [SCN*].

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[Fe^*] = initial # moles of Fe / V_{total}
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{**Note**: while the initial # of moles of Fe does not change from one equilibrium solution to the next, the total volume does change.

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[SCN^*] = \# \text{ moles of SCN } / V_{\text{total}}
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{Note: Both the # of moles of SCN and the total volume will change from one equilibrium solution to the next.}

- c. Calculate [FE*] + [SCN*].
- d. Calculate [FE*] [SCN*].
- e. Calculate A ($[FE^*] + [SCN^*]$).

{**Note:** *A is the Absorbance value for each equilibrium solution.*}

- d. Calculate A / ([FE*] [SCN*]).
- f. Calculate A ([FE*] + [SCN*]) / ([FE*] [SCN*]).
- 3. Create a Beer's Law Plot, using

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x = A([FE^*] + [SCN^*]) / ([FE^*] [SCN^*]).
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- $y = A / [FE^*] [SCN^*]$
- 4. Add a trendline to the plot. Determine the slope of the line to 3 significant digits.
- 5. Determine the equilibrium constant, K_{eq} , for the reaction.
- 6. Determine the concentration of the unknown.
 - a. Make a Beer's Law plot with [KSCN*] concentration on the x-axis & corresponding Absorbances on the y-axis.
 - b. Add a trendline with equation.
 - c. Plug in the absorbance for your unknown into the equation for "y" and solve for your concentration "x".

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Name:		Sect#:	
Lab Partner's Name:	ONLINE	Date:	

Datasheet 1

 $Group \ A-Determination \ of \ Lambda \ Max \ for \ the \ Iron$

Wavelength (nm)	% Transmittance	Absorbance
400	70.2	
425	55	
450	45.8	
475	44.4	
500	50	
525	61	
550	73.6	
575	84.8	
600	92.2	
625	96.8	
650	98.8	
675	99.8	
700	100	

Wavelength at Maximum Al	bsorbance, λ_{max} , nm		_	
Molarity of Fe(NO ₃) ₃ , M	<u>0.0025 M</u>	Molarity of KSCN, M _	<u>0.0025 M</u>	
Vol. of Fe(NO ₃) ₃ , ml	<u>10.00 ml</u>			
Molarity of dil. Fe(NO ₃) ₃ , M				

Experimental Data for Group B:

Soln. #	Vol. KSCN added, ml	% Transmittance	Absorbance
1	5	95.7	
2	10	94.4	
3	15	90.6	
4	20	86.9	
5	25	80.4	
6	30	85.3	
7	35	81.3	
8	40	75.8	
9	45	70.2	
10	50	72.2	
UNK	XXXXXXXXX	61.1	

Name:	Sect#:
Lab Partner's Name:	Date:

Post Lab Questions

- 1. Why is it important that you avoid any loss of solutions when transferring and measuring solutions in this experiment?
- 2. According to the Beer's Law equation, A = abc. Why is it you did not need to know the value for the molar absorptivity constant or the pathlength for the calculations you did in this experiment?
- 3. For each determination of the $\%T \sim 8$ ml of the solution were used. What would have been the effect on your determination of the %T if samples of ~ 9 ml were used instead? Explain briefly.
- 4. Frank and Oswalt report a molar absorptivity of 4700 L/mole cm for the thiocyanatoiron(III) ion. If the given pathlength was 1.00cm, what %T would you expect for a solution that has a concentration of 1.0×10^{-4} M thiocyanatoiron(III) ion?

5. Create a **Beer's Law Plot**, using

$$x = A [FE^*] + [SCN^*] / [FE^*] [SCN^*].$$

 $y = A / [FE^*] [SCN^*]$

Record the following information (slope, K_{eq} and concentration of unknown.)

- a. Add a trendline to the plot. Determine the slope of the line to 3 significant digits.
- b. Determine the equilibrium constant, K_{eq} , for the reaction.
- c. Determine the concentration of the unknown.

- 6. A student mixed 4.00 ml of $1.02 \times 10^{-1} \text{ M}$ Fe(NO₃)₃ with 100.00 ml of $1.98 \times 10^{-4} \text{ M}$ KSCN, using 0.50 M HNO₃ as the solvent for both solutions. He found that the absorbance of the resulting equilibrium mixture to be 0.253. Given this data, calculate the following:
 - a.) [Fe*]
 - b.) [SCN*]
 - c.) [FE*] + [SCN*]
 - d.) [FE*] [SCN*]
 - e.) $A ([FE^*] + [SCN^*])$
 - f.) A / ([FE*] [SCN*])
 - g.) A ([FE*] + [SCN*]) / ([FE*] [SCN*])