Iron(III) Nitrate and Potassium Thiocyanate

Introduction:
Although equilibrium concentrations are very important quantities, they are often difficult to measure. If one of the products we are trying to examine is colored, it becomes significantly easier to determine its concentration in solution by measuring the absorbance of the solution and using Beer's Law.

Safety Concerns:
No specific concerns for this experiment. As always, goggles must be worn and waste disposed of in the designated container.

Experimental Procedure:

I. Reaction between Iron (III) Nitrate and Potassium Thiocyanate

A. Dissolve a small amount (spatula tip) of solid iron (III) nitrate, $Fe(NO_3)_3 \cdot 9H_2O$, in 20 mL of distilled water in a small beaker. Dissolve approximately the same amount of potassium thiocyanate solid, KSCN, in another 20 mL of water. Label each beaker and record the appearance of the solutions.
B. Mix about 10 mL of each of the prepared solutions in a different beaker. Record your observations. Save the remaining portions of the two solutions.
C. Assuming that this is an example of a metathesis reaction involving dissolved ions, which ions are responsible for the colored product you have observed? There is an assortment of different salts containing these ions in the lab. (Please leave the bottles where others can find them.) Using these salts and the saved solutions from the preceding step, design and conduct several experiments that will enable you to conclude which ions are responsible for the formation of the colored complex. Record the results of your experiments. You must have a chemical test that shows whether each of the ions in the experiment $\{Fe^{3+}(aq), NO_3^-(aq), K^+(aq), SCN^-(aq)\}$ is responsible for the observed reaction. Write the net-ionic equation for the reaction assuming a 1:1 stoichiometry for the reactants.

II Determination of the Beer's Law Constant

A. Using pipettes, prepare a number of solutions containing 5.00mL of KSCN stock solution and 15.00 mL of $Fe(NO_3)_3(aq)$ stock solution. The results of the rest of this experiment will depend upon the Beer's Law constant you calculate from these solutions, so prepare as many as necessary to give you confidence in your result. Allow these solutions to react for 5-10 minutes and then record their absorbance vs. wavelength spectra. Choose a wavelength at or near a peak in the spectrum and record in your lab notebook both the wavelength and the absorbance at this wavelength for the solutions you measured.

Q: What is the limiting reagent in these reactions? How will these conditions affect the equilibrium compared to a reaction that had no limiting reactant?

B. Assuming the limiting reactant is completely consumed to form the desired 1:1 product in these samples, calculate the value of "$\varepsilon \bullet l$" in Beer's Law at the wavelength you chose. *Include error in your results.*

Q: Is it a valid assumption that the limiting reactant is completely consumed in this reaction? If so, what does this tell you about the magnitude of the equilibrium constant for this reaction? As an example, would this assumption be valid if the equilibrium constant was very small? Very large? Explain.

III. Exploring the equilibrium quantitatively

A. Obtain two burettes and label one as water and one as $Fe(NO_3)_3$. Make sure the burettes are clean and they do not leak. In clean labeled beakers, take about 50 mL each of the stock KSCN and $Fe(NO_3)_3$ solutions. Rinse each burette with several 3-5 mL portions of the solution that will be used in them subsequently. Fill the burettes with the respective solutions. Make sure that the stopcock and the burette tip do not have any air bubbles in them. If you have any questions, ask for help ***before*** you continue to prepare your samples.

B. You will be preparing at least 8 solutions (you may prepare more if you like), each with a total volume of *approximately* 20 mL. Prepare each sample in a clean ***dry*** large test tube. Each solution will contain 5.00 mL of KSCN solution {pipette}, **0.50-8.00 mL of $Fe(NO_3)_3(aq)$ (cover this entire range of volumes for $Fe(NO_3)_3$) {burette}**, and enough water {burette} to make approximately 20 mL of solution. For example, one of your samples might contain 5.00 mL KSCN(aq), 6.13 mL $Fe(NO_3)_3(aq)$, and 8.94 mL of water, for a total solution volume of 20.07 mL. It is not necessary for the total volume to be exactly 20.00 mL, but it *is* important that you know the total (additive) volume of the solution. Your 8 samples should cover the full range of volumes of the iron solution listed above. **If you report all of your solutions with a total volume of 20.00 mL, you are not using the equipment correctly and will lose points for this experiment.**

C. Mix your samples thoroughly and allow them to react for 5-10 minutes before recording the spectra of each solution at the wavelength of maximum absorbance determined in part IIA. It is easiest to mix these solutions by covering the tube (rubber stopper or Parafilm) and *inverting the tube several times*.

Q: What is the limiting reagent in each of these samples? If you make the same assumption as in Part IIB above, what would you expect the absorbance of each solution to be? Is this what you observe? What does this observation tell you about the magnitude of the equilibrium constant for this reaction? It may be helpful to look at these data graphically. Prepare a plot of absorbance vs. initial iron(III) concentration. Explain any trend you see in your data.

D. Calculate the initial concentrations {after mixing but before reacting} of the ions responsible for the observed reaction for each of the samples you have prepared. Using the value of "ε•l" calculated above, calculate the equilibrium concentration of the colored product in each of your samples. Once you know the initial concentration of each reactant and the equilibrium concentration of product, you should be able to calculate the equilibrium concentrations of all species and calculate the equilibrium constant for the reaction.

Q: Is the value of the equilibrium constant consistent with your predictions earlier in the experiment? Explain any deviations.