Inorganic Synthesis

Inorganic complexes are metal ions bound to and surrounded by ligands. Ligands are small molecules, ions or larger organic molecules. Many of the important enzymes and energy-producing molecules in living organisms are inorganic complexes. For example, hemoglobin is an iron complex and chlorophyll is a magnesium complex. Very often complexes are colored, which adds to their interest. In this experiment, you will synthesize and purify an inorganic complex.

Potassium trioalatoiron (III)

Iron (III) chloride, a source of the iron (III) ion, and potassium oxalate will be mixed together to form potassium trioalatoiron (III). In addition to the two basic ingredients, water will be used. Water, or some other solvent, is often used in syntheses because it speeds up the reaction. Solids tend to react very slowly with each other because they only contact each other at their surfaces. Molecules inside the solid can’t reach anything to react with. Dissolved molecules or ions are all able to find a reactive partner. Sodium acetate will also be added, because iron (III) ions will react with water to make rust. Acetate ions make a water-soluble complex with iron (III) ions that is resistant to forming rust.

The chemical equation is:

\[
\text{FeCl}_3 \cdot 6\text{H}_2\text{O}_{(s)} + 3\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}_{(s)} \rightarrow \text{K}_3\text{Fe}((\text{C}_2\text{O}_4)_3 \cdot 3\text{H}_2\text{O}_{(s)} + 3\text{KCl}_{(aq)} + 6\text{H}_2\text{O}_{(l)}
\]

A common strategy in inorganic synthesis is to synthesize an insoluble compound from soluble starting materials. This effectively separates the product from the reactants and makes it easy to isolate the product by filtration.

Purification of the product will be performed through recrystallization, which is simply dissolving a solid in hot solvent and letting it precipitate as the solvent cools off. Often
impurities in the product, such as small amounts of starting materials, will not precipitate from the cool solvent.

**Procedure**

Mix 5.0 g NaC$_2$H$_3$O$_2$·3H$_2$O with 10 mL of water. In a separate beaker, dissolve 4.0 g FeCl$_3$·6H$_2$O in 10 mL of water. Mix the two solutions together. Dissolve 12.0 g of potassium oxalate (K$_2$C$_2$O$_4$·H$_2$O) in 20 mL of hot water. Add the oxalate solution to the iron solution, stir for 15 minutes, and then cool to ice temperature.

Isolate the solid by vacuum filtration and recrystallize the solid from water. To recrystallize, put the solid in a 100 mL beaker and add a small amount of water (about 5 mL). Heat the solution on a hot plate. Once the water begins to boil, check to see if the solid has dissolved. If not, add more water and allow it to reach boiling temperature again. The goal is to use the minimum amount of hot water to dissolve the sample. Once the sample has dissolved, remove the flask from the hotplate and decant the solution from any remaining solids.

Cool the solution to 0º C, and filter to collect the crystals. After the crystals have been isolated on the filter paper, rinse them with cold methanol. The methanol will dissolve and wash away any water left on solid, allowing them to dry faster. Let air pass through the solid to evaporate the methanol for 15 minutes. Weigh the dried product.

Questions:

1. Calculate the percent yield of the synthesis.

2. Estimate the wavelength of light that would be most strongly absorbed by a solution of your product. Explain your answer.

3. Detail a process through which you could determine the density of your crystals.