Calcium Iodate

Introduction:

A common method for purification and isolation of various substances involves the precipitation of either the desired substance or impurities from an impure mixture. For this method to work, it is desirable that all of the desired precipitate forms and can be removed from the mixture, but this is not typically the case. It is much more common that some of the "precipitate" remains dissolved in the solvent, meaning that pure compounds can be difficult to obtain in high yield in this manner.

Safety Concerns:

There are no specific concerns for this experiment. Wear goggles at all times and dispose of all waste in the appropriate container.

Experimental Procedure:

I. Precipitation Reaction between Calcium Nitrate and Potassium Iodate

Using appropriate graduated cylinders, prepare the following mixtures of $Ca(NO_3)_2$ and KIO_3 in clean labeled 150 mL beakers.

Solution #1: 25.0 mL $Ca(NO_3)_2$ + 25.0 mL KIO_3

Solution #2 15.0 mL H_2O + 10.0 mL $Ca(NO_3)_2$ + 25.0 mL KIO_3

Mix each solution with a separate stirring rod. Do not remove the stirring rods from the solution or you may lose some of the solid that should be recovered and weighed later on. If solid is not forming, you may try scratching the sides and bottom of the beaker with the stirring rod to help induce crystallization. Let these solutions stand for at least 15 minutes. Stir occasionally.

The following questions are designed to help you interpret your data. It may be helpful for you to answer these questions while you are in the lab and can interact with the instructor but you are not required to record the answers to these questions in your lab notebook.

Q: In what ways are these two solutions different? What role does the water play in the second solution? Determine the limiting reactant and theoretical yield of solid for each of the solutions.

II. Experimental Mass of the Precipitates

Label two filter paper circles with pencil. Weigh each of the filter papers. Following the directions given by your lab instructor, filter each solution. When the filtration has been accomplished, set the filtrates aside for use in part III. ***DO NOT THROW AWAY YOUR FILTRATES.*** Rinse the precipitates with methanol in the fume hood to help remove residual liquid from the solids. Remove the filter papers from the funnels and place them on watch glasses. Dry in air for several minutes and then place them in a 90-110ºC drying oven for at least 45 minutes. **Allow the samples to cool to room temperature** before recording their final mass. Perform part III while your samples are drying in the oven.

Q: What was your percent yield for each sample? Why was it not 100%?

III. Qualitative testing of the filtrate

A. Standards - Characteristic tests for the presence of Ca^{2+} and IO_3^-. The purpose of these tests is to see what happens with (relatively) high concentration of the test ion, relatively low concentrations of the test ion, and with no test ion present. Make careful observations so you can correctly interpret the results of these tests on your filtrates in Part B.

1. Qualitative Tests for Ca^{2+}

$$3\,Ca^{2+}(aq) + 2\,PO_4^{-3}(aq) \leftrightarrows Ca_3(PO_4)_2(s)$$

   a. Obtain about 1 mL of $Ca(NO_3)_2$ solution in a small test tube. Add about 0.5 mL (10 drops) of Na_3PO_4 solution. This is a *strong positive* test for $Ca^{2+}(aq)$.
   b. Obtain 1 drop of $Ca(NO_3)_2$ solution in a small test tube and add about 1 mL of water. Add about 0.5 mL (10 drops) of Na_3PO_4 solution. This is a *weak positive* test for $Ca^{2+}(aq)$.
   c. Repeat this test with 1mL of water but no added $Ca(NO_3)_2$ solution.

Q: Are your observations different? Why?

2. Qualitative Tests for IO_3^-

$$IO_3^-(aq) + 5\,I^-(aq) + 6\,H^+(aq) \leftrightarrows 3\,I_2(aq) + H_2O(l)$$

   a. Obtain about 1 mL of KIO_3 solution in a small test tube. Dissolve a small amount of KI (about the size that would fit on a match head) in the solution and then add about 1 mL of 1 M HCl. This is a *strong positive* test for $IO_3^{-1}(aq)$.
   b. Obtain 1 drop of KIO_3 solution in a small test tube and add about 1 mL of water. Dissolve a small amount of KI (about the size that would fit on a match head) in the solution and then add about 1 mL of 1 M HCl. This is a *weak positive* test for $IO_3^-(aq)$.
   c. Repeat this test with 1mL of water but no added KIO_3 solution.

Q: Are your observations different? Why?

B. Filtrates (Note that in this section you will perform a total of 4 tests, 2 on each filtrate.)

1. Test both your filtrates for the presence of Ca^{2+}. Perform the same test for Ca^{2+} as above (1a), but instead of the known Ca^{2+} solution, use about 1 mL of filtrate.
Note: Hold the tube against the dark background. Any presence of cloudiness is a positive result.

Q: Was calcium present in your filtrates? If so, based upon your observations, how much Ca^{2+} was present (a significant amount or just a little bit)? Are these observations consistent with the identity of the limiting reagent in each solution?

2. Test both your filtrates for the presence of IO_3^-. Perform the same test for IO_3^- as above (2a), but instead of the known IO_3^- solution, use about 1 mL of filtrate. Any presence of cloudiness is a positive result.

Q: Was iodate present in your filtrates? If so, based upon your observations, how much IO_3^- was present (a significant amount or just a little bit)? Are these observations consistent with the identity of the limiting reagent in each solution?

Record your results table form as suggested by your instructor.

Review all sections of this experiment. We often describe salts as being either "soluble" or "insoluble". For example, you might say "Sodium chloride is soluble" or "Calcium carbonate is insoluble." Given your observations throughout this experiment, why do these statements not completely describe the concept of "solubility"?