Precipitation Reactions and Percent Yield – Procedure

Purpose:

In this experiment, the precipitation reaction between cobalt(II) nitrate and sodium phosphate is performed, and the percent yield of the reaction is calculated.

Procedure:

Specific reaction mixtures formulated in Table 1 will be assigned to each group. In each case, a precipitate is formed upon mixing $Co(NO_3)_2(aq)$ and $Na_3PO_4(aq)$. The precipitate will be collected by filtration, washed, dried, and weighed.

- In those mixtures where ***$Co(NO_3)_2$ is the limiting reagent***, the moles of precipitate will be related to the moles of $Co(NO_3)_2$ originally present, by the balanced reaction stoichiometry, and the filtrate will be colorless.
- In those mixtures where ***Na_3PO_4 is the limiting reagent***, the moles of precipitate will be related to the moles of Na_3PO_4 originally present, by the balanced reaction stoichiometry. These mixtures will contain an excess of $Co(NO_3)_2$, and the filtrate will be pink/red due to the presence of the red cobalt(II) ion (Co^{2+}) remaining in solution.

Each group will be responsible for preparing three solutions from Table 1 as assigned by your instructor. Each group will determine the mass of precipitate formed in each case as well as the color of the filtrate (remaining solution). For each of the three solutions assigned to your group, the limiting reagent must be determined mathematically, and the percent yield of precipitate calculated. The mathematically determined limiting reagent should agree with the color of the filtrate, as described above.

Table 1.

solution number	mL of $Na_3PO_4(aq)$	mL of $Co(NO_3)_2(aq)$
1	10.0	2.0
2	10.0	3.0
3	10.0	4.0
4	10.0	6.0
5	10.0	7.0
6	10.0	8.0
7	10.0	10.0
8	10.0	11.0
9	10.0	12.0

The Precipitation Reaction

Your instructor will assign a single group letter, A-L, to you and your lab partner. Each group letter has three correlating solution numbers according to the table below. For instance, group A will perform three trials of the precipitation reaction, using the quantities given in solution numbers 1, 5, and 8.

	group											
solution # (Table 1)	A	B	C	D	E	F	G	H	I	J	K	L
1	●			●				●		●		
2		●					●		●		●	
3			●			●				●		●
4				●	●						●	
5	●				●			●	●			●
6			●			●	●					
7		●					●				●	
8	●	●			●			●		●		●
9			●	●		●			●			

Clean three beakers (100 mL, 150 mL, or 250 mL). Label each beaker with the number of each one of your three solutions. To each of the three labeled beakers, pipette 10.0 mL of the Na_3PO_4 solution, and then pipette the appropriate volume of $Co(NO_3)_2$ solution. The best way to do this is to rinse the pipette with a little of the Na_3PO_4 solution, then pipette the 10.0 mL into each of the beakers. Next rinse the pipette with deionized water and then with a little of the $Co(NO_3)_2$ solution. Finally, pipette the required volumes of the $Co(NO_3)_2$ solution into each beaker. Rinse the pipette with deionized water before you return it. Swirl each beaker to thoroughly mix the reagents and form the precipitate. Make sure to record in your notebook the concentrations of both the Na_3PO_4 and $Co(NO_3)_2$ solutions.

In a fume hood, set up a vacuum filtering apparatus using your Buchner funnel, filter adapter, filtering flask, and rubber hose, and securely clamp it to a ring stand (Figure 1).

Figure 1. Vacuum filtration setup
(a) Buchner funnel
(b) filter adapter
(c) filter flask

Check your filter flask for visible cracks or star cracks. If your flask is cracked, discard it and obtain a new flask from the stockroom. Attach one end of the thick-walled rubber hose to the

side arm of the filter flask and attach the other end to the yellow house-vacuum inlet in the fume hood. The use of vacuum, versus gravity alone, will speed the filtration and isolation of the precipitate.

Obtain three pieces of Whatman #4 filter paper, sized specifically for your Buchner funnel (4.25 cm diameter), and with a pencil, label them with the numbers of your solutions. Also label three watch glasses with the numbers of your solutions. Place each piece of filter paper onto its corresponding watch glass, and record the combined mass of each watch glass/paper pair. Be certain that the balance is set to zero grams before weighing. Place one of the pieces of filter paper in the Buchner funnel and fully turn on the vacuum valve. Make sure the filter paper is sitting flat, secure, and covers all of the holes in the funnel.

Pour the gelatinous precipitate from the corresponding numbered solution into the Buchner funnel. Allow the vacuum to pull through the liquid filtrate until you are left with only the solid precipitate in the Buchner funnel. Rinse the reaction beaker with no more than 5 mL of deionized water, dislodging any precipitate which remained in the beaker with your rubber policeman. Transfer this first rinse to the Buchner funnel and again allow the vacuum to pull through the liquid phase. Repeat this rinsing process two more times, for a total of three rinses. The purpose of these rinses is two-fold:

1. *completely* transfers the precipitate to the Buchner funnel. The yield is an important part of your grade so work carefully.
2. washes the precipitate of excess, unreacted aqueous reagent. If not removed, this will falsely add to the mass of your dried precipitate.

Finish rinsing the precipitate by adding two 5 mL portions of acetone. Add the first 5 mL portion of acetone to the Buchner funnel and allow it to be pulled through before you add the second portion. Acetone is a volatile (low boiling point, evaporates easily, do not breathe the vapors) organic solvent which is soluble in water. The two acetone rinses will wash away the excess water, allowing the precipitate to dry faster and dry to the correct mass.

*** **Acetone is extremely flammable!** ***

Absolutely no Bunsen burner use should
occur during this entire laboratory experiment!

Once the second portion of acetone has been pulled through, let the vacuum pull air through the sample for a minute or two. During this time you may notice the precipitate lighten in color and it may begin to crack and pull away from the walls of the funnel. Pulling air through will help to dry the precipitate and also aid in its removal from the funnel.

Remove the funnel from the filter flask and turn off the vacuum. Using a spatula, pry the filter paper from the bottom of the Buchner funnel. Lift the paper and precipitate out of the funnel and place it on the correspondingly labeled watch glass. If you are careful, you can remove the paper and precipitate as an entire "cake". Scrape any remaining precipitate from the walls of the Buchner funnel and add this to your watch glass. Record the color of the filtrate (filtered solution) in your notebook.

Waste Disposal: Dispose of the liquid filtrate in the appropriate liquid waste container.

Repeat the entire vacuum filtration/isolation procedure for the two remaining precipitates. The filtration and isolation of all three precipitates will require approximately *60 minutes* to complete. Come to lab prepared and make sure to give yourself enough time to properly isolate all three precipitates without rushing through or skipping steps. Carefully store all three watch glasses in your drawer and leaved them there for one week to air dry.

The Dried Precipitates

Now that the precipitates have been dried, weigh the precipitate-watch glass-filter paper combinations and record the masses in your notebook.

Waste Disposal: Dispose of the dried precipitate and the filter paper in the appropriate solid waste container.

Results: For each trial, report the limiting reagent, the reagent in excess, actual yield of the precipitate, theoretical yield of the precipitate, and percent yield.

Conclusion:

Your conclusion must be a well written synopsis that addresses the experimental purpose and restates the results. Did the color of the filtrate agree with the calculated limiting reagent? What is the most desirable percent yield and how do your values from each trial compare? Suggest sources for possible errors that could have affected your percent yields. Include the balanced overall chemical reaction as well as the net ionic equation as part of your conclusion.