EXPERIMENT

Precipitating Calcium Phosphate

OBJECTIVES

- To determine the percent yield of calcium phosphate from a precipitation reaction.
- To determine the percentage of calcium chloride in an unknown mixture.
- To gain proficiency in transferring and filtering a precipitate.

DISCUSSION

In this experiment, we will use aqueous solutions to produce a precipitate of insoluble calcium phosphate, $Ca_3(PO_4)_2$. The equation for the reaction is

$$3\ CaCl_2(aq) + 2\ Na_3PO_4(aq) \rightarrow Ca_3(PO_4)_2(s) + 6\ NaCl(aq)$$

The precipitate will be collected in filter paper, which separates the insoluble particles from aqueous solution. The experimental mass of the precipitate is referred to as the **actual yield**. The calculated mass of the precipitate using the above equation is referred to as the **theoretical yield**.

The **percent yield** from a chemical reaction is an expression for the amount of actual yield compared to the theoretical yield. While some experimental errors lead to high results, other errors may give low results. Thus, the percent yield can be greater than—or less than—100%.

Percent Yield of Calcium Phosphate from Calcium Chloride

Example Exercise 1 • % Yield of $Ca_3(PO_4)_2$ from $CaCl_2$

A 0.555-g sample of calcium chloride gives a 0.525-g precipitate of calcium phosphate. Calculate the theoretical yield and percent yield of $Ca_3(PO_4)_2$.

Solution: According to the balanced chemical equation, 3 mol $CaCl_2$ (110.98 g/mol) produce 1 mol $Ca_3(PO_4)_2$ (310.18 g/mol) precipitate. We can calculate the theoretical mass of precipitate as follows:

$$0.555\ \cancel{\text{g } CaCl_2} \times \frac{1\ \cancel{\text{mol } CaCl_2}}{110.98\ \cancel{\text{g } CaCl_2}} \times \frac{1\ \cancel{\text{mol } Ca_3(PO_4)_2}}{3\ \cancel{\text{mol } CaCl_2}} \times \frac{310.18\text{ g } Ca_3(PO_4)_2}{1\ \cancel{\text{mol } Ca_3(PO_4)_2}}$$

$$= 0.517\text{ g } Ca_3(PO_4)_2$$

Since the actual yield of $Ca_3(PO_4)_2$ is 0.525 g, the percent yield is

$$\frac{\text{actual yield}}{\text{theoretical yield}} \times 100\% = \%\text{ yield}$$

$$\frac{0.525\ \cancel{\text{g}}}{0.517\ \cancel{\text{g}}} \times 100\% = 102\%$$

Percentage of Calcium Chloride in an Unknown Mixture

When an unknown mixture containing calcium chloride reacts with sodium phosphate, the equation for the reaction is the same as above. In this calculation, however, we will relate the mass of calcium phosphate to the mass of calcium chloride in the original unknown mixture.

Example Exercise 2 • % $CaCl_2$ in an Unknown Mixture

A 1.000-g unknown mixture containing calcium chloride gives a 0.565-g precipitate of calcium phosphate. Calculate the percentage of $CaCl_2$ in the unknown mixture.

Solution: In this example, we must relate the mass of precipitate product to the mass of the original $CaCl_2$ reactant.

$$0.565\ \cancel{\text{g } Ca_3(PO_4)_2} \times \frac{1\ \cancel{\text{mol } Ca_3(PO_4)_2}}{310.18\ \cancel{\text{g } Ca_3(PO_4)_2}} \times \frac{3\ \cancel{\text{mol } CaCl_2}}{1\ \cancel{\text{mol } Ca_3(PO_4)_2}} \times \frac{110.98\text{ g } CaCl_2}{1\ \cancel{\text{mol } CaCl_2}}$$

$$= 0.606\text{ g } CaCl_2$$

If the sample mixture has a mass of 1.000 g, the percentage of $CaCl_2$ is

$$\frac{\text{mass } CaCl_2}{\text{mass sample}} \times 100\% = \%\ CaCl_2$$

$$\frac{0.606\ \cancel{\text{g}}}{1.000\ \cancel{\text{g}}} \times 100\% = 60.6\%$$

EQUIPMENT and CHEMICALS

- 250-mL beaker
- 100-mL graduated cylinder
- ring stand
- wire gauze
- clay triangle
- 400-mL beaker
- wash bottle with distilled water
- glass stirring rod + rubber policeman
- calcium chloride, anhydrous $CaCl_2$
- sodium phosphate solution, 0.5 *M* Na_3PO_4
- filter paper
- long-stem funnel (75 mm diameter)
- unknown calcium chloride mixtures (50–70% $CaCl_2$)

PROCEDURE

A. Percent Yield of $Ca_3(PO_4)_2$ from $CaCl_2$

1. Place a 250-mL beaker on the balance, and record the mass. Add about 0.5 g of calcium chloride, and reweigh accurately.

2. Dissolve the $CaCl_2$ sample completely in 50 mL of distilled water. Using a graduated cylinder, add 10 mL of 0.5 *M* Na_3PO_4 solution to the sample in the 250-mL beaker.

3. Support the beaker with a wire gauze on a ring stand. Bring the solution to a gentle boil and then turn off the burner. Allow the precipitate to digest until the solution is cool.

   Note: As the precipitate settles from aqueous solution, add a few drops of Na_3PO_4 solution to test for completeness of precipitation. If the clear solution becomes cloudy, add more Na_3PO_4 solution to assure that all of the calcium has been precipitated from solution.

4. Weigh a disk of filter paper. Prepare a filter paper cone by folding the disk twice. Insert the filter paper into the funnel, and moisten with distilled water using the wash bottle.

5. Assemble a filtering apparatus as shown in Figure 1.

6. Without disturbing the precipitate, carefully pour off the supernate into the filter paper, using a stirring rod to guide the flow as shown in Figure 1(a). Rinse out the bulk of the precipitate with a stream of water from the wash bottle as shown in Figure 1(b). Clean the beaker using a rubber policeman and rinse the residue into the filter paper.

   Note: The precipitate is "jelly-like" and filters quite slowly. To speed filtration, first pour off the clear supernate. Then transfer the precipitate using a minimum amount of wash water. To avoid delay, allow the filtration to continue and begin the unknown part of the experiment.

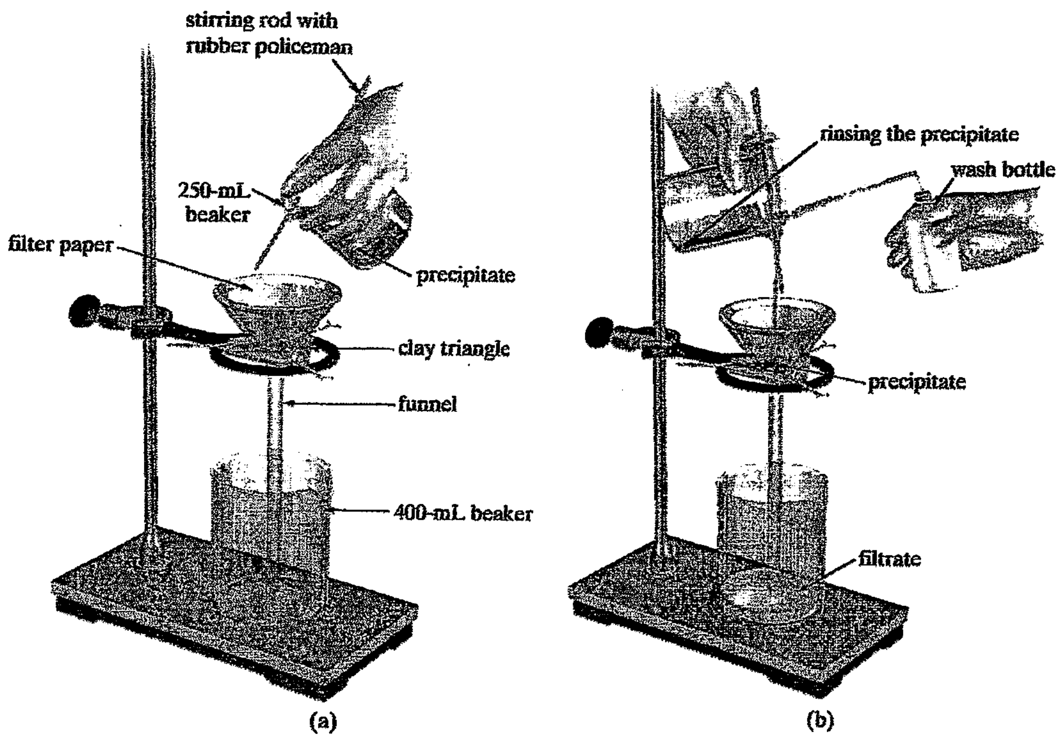

Figure 1 Apparatus for Filtration (a) When the precipitate settles, pour off the supernate. (b) After the supernate passes through the filter paper, rinse the precipitate into the filter paper using a stream of water from a wash bottle.

7. After the supernate has passed through the filter, *carefully* remove the paper cone from the funnel. After the precipitate is completely dry, weigh the filter paper and precipitate.

   Note: If an oven is available, dry the precipitate overnight at ~110°C.

8. Calculate the theoretical yield of calcium phosphate from the mass of the calcium chloride. Find the percent yield.

B. Percentage of $CaCl_2$ in an Unknown Mixture

1. Obtain an unknown sample mixture containing calcium chloride, $CaCl_2$. Record the unknown number in the Data Table.

2. Place a 250-mL beaker on the balance and record the mass. Add about 1 g of unknown mixture, and reweigh accurately.

3. Repeat steps 1–7 as in Procedure A; substitute the unknown mixture for calcium chloride.

4. Calculate the mass of calcium chloride in the unknown sample from the mass of precipitate. Find the percentage of calcium chloride in the unknown mixture.

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PRELABORATORY ASSIGNMENT*

1. In your own words, define the following terms:

   actual yield

   digestion

   filtrate

   percent yield

   supernate

   theoretical yield

2. What difficulty arises if the precipitate is not allowed to settle completely from solution?

3. What is the purpose of the rubber policeman?

4. What should be done if particles of precipitate appear in the filtrate?

5. Is it possible to have a percent yield of calcium phosphate that is greater than 100%?

6. A sample of calcium chloride reacts with sodium phosphate to give a precipitate of calcium phosphate. Refer to Example Exercise 1 and determine the theoretical yield of calcium chloride, $CaCl_2$, from the following data:

mass of beaker + $CaCl_2$	100.621 g
mass of beaker	100.111 g
mass of $Ca_3(PO_4)_2$ ppt	0.466 g

What is the percent yield of calcium chloride, $CaCl_2$?

7. A 0.995-g unknown mixture containing calcium chloride reacts to give 0.505 g precipitate of calcium phosphate. Refer to Example Exercise 2 and find the mass of calcium chloride, $CaCl_2$, in the unknown mixture from the following data:

mass of beaker + unknown mixture	105.020 g
mass of beaker	104.025 g
mass of $Ca_3(PO_4)_2$ ppt	0.505 g

What is the percentage of calcium chloride, $CaCl_2$, in the unknown mixture?

8. What are the major sources of error in this experiment?

9. What safety precautions must be observed in this experiment?

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DATA TABLE

A. Percent Yield of $Ca_3(PO_4)_2$ from $CaCl_2$

mass of beaker + $CaCl_2$	__________ g
mass of beaker	__________ g
mass of $CaCl_2$	__________ g
mass of filter paper + $Ca_3(PO_4)_2$ ppt	__________ g
mass of filter paper	__________ g
mass of $Ca_3(PO_4)_2$ ppt *(actual yield)*	__________ g

Show the calculation for theoretical yield of $Ca_3(PO_4)_2$ for trial 1 (see Example Exercise 1).

mass of $Ca_3(PO_4)_2$ ppt *(theoretical yield)* __________ g

Show the calculation for percent yield of $Ca_3(PO_4)_2$ for trial 1 (see Example Exercise 1).

Percent Yield of $Ca_3(PO_4)_2$ __________ %

B. Percentage of $CaCl_2$ in an Unknown Mixture UNKNOWN # ______

mass of beaker + unknown mixture ____________ g

mass of beaker ____________ g

mass of unknown mixture ____________ g

mass of filter paper + $Ca_3(PO_4)_2$ ppt ____________ g

mass of filter paper ____________ g

mass of $Ca_3(PO_4)_2$ ppt ____________ g

Show the calculation for the mass of $CaCl_2$ in the unknown mixture for trial 1 (see Example Exercise 2).

mass of $CaCl_2$ ____________ g

Show the calculation for the percentage of $CaCl_2$ in the unknown mixture for trial 1 (see Example Exercise 2).

Percentage of $CaCl_2$ ____________ %

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POSTLABORATORY ASSIGNMENT

1. A 0.914-g sample of stannous fluoride gives a 1.078-g precipitate of stannous phosphate. Calculate the theoretical yield and percent yield of $Sn_3(PO_4)_2$.

$$SnF_2(aq) + Na_3PO_4(aq) \rightarrow Sn_3(PO_4)_2(s) + NaF(aq)$$

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2. A 10.000-g sample of toothpaste containing stannous fluoride gives a 0.145-g precipitate of stannous phosphate. What is the percentage of SnF_2 in the toothpaste sample?

$$SnF_2(aq) + Na_3PO_4(aq) \rightarrow Sn_3(PO_4)_2(s) + NaF(aq)$$

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3. Marble is composed mainly of calcium carbonate, $CaCO_3$. If 0.750 g of marble reacts with hydrochloric acid to produce 165 mL of carbon dioxide gas at STP, what is the percentage of calcium carbonate in the marble sample?

$$CaCO_3(s) + HCl(aq) \rightarrow CaCl_2(aq) + H_2O(l) + CO_2(g)$$

4. Tooth enamel is mainly hydroxyapatite, $Ca_{10}(PO_4)_6(OH)_2$. If 1.150 g of tooth enamel reacts with hydrochloric acid to give 1.231 g of calcium chloride, what is the percentage of hydroxyapatite in the tooth enamel sample?

$$Ca_{10}(PO_4)_6(OH)_2(s) + HCl(aq) \rightarrow CaCl_2(aq) + H_3PO_4(aq) + H_2O(l)$$

5. (optional) Milk of magnesia, $Mg(OH)_2$, is prepared from the reaction of aqueous magnesium sulfate and sodium hydroxide solution. If a solution containing 125 g of $MgSO_4$ is added to a solution with 115 g of NaOH, what is the mass of milk of magnesia produced?

$$MgSO_4(aq) + NaOH(aq) \rightarrow Mg(OH)_2(s) + Na_2SO_4(aq)$$