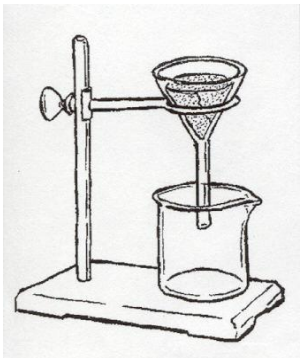
4 Synthesis and Analysis Of Calcium Sulfate

Version: September 9, 2008

- **See by your own measurements how the amount of a single reaction-the limiting reactant-determines the maximum amount of product that can be produced.**
- **See by your own measurements how other factors cause the actual yield to be less than the maximum.**
- **Determine the identity of a product by chemical analysis.**



Filter Paper in Funnel

DISCUSSION

When solutions of sodium sulfate and calcium chloride react they produce an insoluble precipitate of calcium sulfate.

$$Na_2SO_4(aq) + CaCl_2(aq) + xH_2O(l) \rightarrow CaSO_4 \cdot xH_2O(s) + 2NaCl(aq)$$

In this experiment you will mix solutions of the reacts and collect the precipitated calcium sulfate by filtration. The amount of solid that can be theoretically collected is determined by the reactant that is used up first—the limiting reactant. Once the limiting reactant has reacted completely, the net reaction stops. In practice, the actual amount of product collected is less than the calculated amount. The % yield is defined by the following equation:

$$\%yield = \frac{Actual\ Yield\ (from\ experiment)}{Theoretical\ yield\ (from\ calculation)} \times 100\%$$

The percent yield is a function of the skill of the experimenter and the efficiency of the reaction. Yields less than 100% are not necessarily the fault of the experimenter.

Many solid compounds exist as hydrates. These compounds have water chemically bound within the solid structure and do not obviously look wet. Solid calcium sulfate occurs naturally in the following three forms differing in the number of water molecules bound within the crystal structure: anhydrite ($CaSO_4$), plaster of paris ($CaSO_4 \cdot \frac{1}{2}H_2O$), and gypsum ($CaSO_4 \cdot 2H_2O$). When calcium sulfate precipitates from solution, any one of these might form. To determine which one is produced in this reaction, it will be carefully dried at low temperature to remove the superficial water that makes the solid wet. Later it will be analyzed to determine how much water is chemically bound within the solid. From this analysis you can determine which of the three forms of calcium sulfate you have collected.

PROCEDURE

A. Preparation and Collection of Calcium Sulfate

1. Weigh a small test tube to the nearest 0.001 g. Label this #1.
2. Weigh a second small test tube and label this #2.
3. To the first test tube, add about 300 mg of sodium sulfate from its color-coded vial (set the syringe at 0.2 *cc*). Determine the mass of the solid to the nearest milligram.
4. To the second test tube, add about 300 mg of calcium chloride dihydrate ($CaCl_2 \cdot 2H_2O$) from the other-colored vial (set its syringe at 0.3 *cc*). Determine the mass of the solid to the nearest milligram.

Add about 3 mL of distilled water to each test tube to dissolve the solids. To speed dissolution of the solid, agitate the solution occasionally by "drumming" your fingers against the side of the tube near the bottom.

5. When both solids have dissolved completely, pour the solution in one test tube into the other, and rinse the empty tube with two 1 mL portions of purified water. Add the rinsings to the mixed solution. Agitate the solution to mix the chemicals. Allow the solution to sit for at least 10 minutes. Record your observations.
6. Weigh and record the mass of a piece of filter paper. Then, fold it in fourths, and

place it in a funnel apparatus as shown in the diagram. Wet the paper with water from your wash bottle so that the paper sticks to the funnel.

Pour the contents of the test tube, in portions, through the filter. Take care not to allow the apparatus to overflow. Rinse the test tube twice with distilled water, and pour the rinsings through the filter.

Remove the filter paper from the funnel, and place it, unfolded, on a watch glass. Place the watch glass and filter paper in your drawer and allow it to air-dry for a week.

Second Week

7. When the filter paper and precipitate are thoroughly dry, measure their mass.

8. Calculate the mass of the calcium sulfate that was collected.

B. Analysis of Product for Water

9. Obtain a special micro crucible and cover from the stockroom. Place the crucible in the holder provided, and heat the crucible and cover strongly for 2 minutes. Allow the crucible and cover to cool for at least 5 minutes. Determine the mass of the cooled crucible and cover to the nearest milligram.

10. Transfer as much of the dry calcium sulfate as conveniently possible to the weighed crucible. Reweigh the crucible, cover and calcium sulfate to the nearest milligram.

11. Strongly reheat the crucible, cover and calcium sulfate for 2 minutes. Allow it to cool for at least 5 minutes. When the crucible is cool, reweigh the crucible, cover and anhydrous calcium sulfate.

12. Calculate the mass of anhydrous calcium sulfate in the crucible.

13. Calculate the moles of anhydrous calcium sulfate ($CaSO_4$)in the crucible.

14. Calculate the mass of water that escaped when the crucible and calcium sulfate were heated.

15. Calculate the moles of water that escaped.

16. Calculate the ratio of moles of water, initially in the sample, to moles of anhydrous calcium sulfate.

17. Write the formula for the hydrate of calcium sulfate that was placed in the crucible.

Which hydrate of calcium sulfate was produced in the precipitation reaction: anhydrite, plaster of paris or gypsum?

C. Calculation of Percent Yield

18. Calculate the mass of sodium sulfate in the original test tube (#1)

19. Calculate the mass of calcium chloride dihydrate in the original test tube (#2).

20. From the initial mass of sodium sulfate, calculate the mass of calcium sulfate compound that would be produced. (You will need to insert the proper value of x in the equation at the beginning of the lab instructions.)

21. From the initial mass of calcium chloride dihydrate, calculate the mass of calcium sulfate compound that would be produced.

22. Identify the limiting reactant.

23. Identify the theoretical yield of calcium sulfate compound.

24. Using the actual yield of calcium sulfate collected in the precipitation reaction and the theoretical yield, calculate the percent yield.

4 Calcium Sulfate

Name _____
Partner _____
Section _____Locker ____
Instructor _____

Enter the data or answer the questions *according to the corresponding step in the procedure*

A. Preparation and Collection of Calcium Sulfate

1. Mass of Test Tube 1	
2. Mass of Test Tube 2	
3. Mass of Test Tube 1 and Na_2SO_4	
4. Mass of Test Tube 2 and $CaCl_2 \cdot 2H_2O$	
5. Observations upon mixing the two solutions	

6. Mass of the filter paper	
7. Mass of dry product and the filter paper	
8. Actual yield of $CaSO_4 \cdot xH_2O$	

CALCULATIONS

B. Analysis of the Product for Water

9. Mass of empty crucible and cover	
10. Mass of crucible, cover and $CaSO_4 \cdot xH_2O$	
11. Mass of crucible, cover and $CaSO_4$ after heating	
12. Mass of $CaSO_4$ in crucible	
13. Moles of $CaSO_4$ in crucible	
14. Mass of H_2O that escaped from $CaSO_4 \cdot xH_2O$	
15. Moles of H_2O that escaped from $CaSO_4 \cdot xH_2O$	
16. The ratio, *x*: moles of water that escaped over moles of anhydrous $CaSO_4$	
17. Formula and name of the hydrate	

C. Calculation of Percent Yield

18. Mass of Na_2SO_4	
19. Mass of $CaCl_2 \cdot 2H_2O$	
20. Expected yield assuming Na_2SO_4 is the limiting reactant	
21. Expected yield assuming $CaCl_2 \cdot 2H_2O$ is the limiting reactant	
22. The limiting reactant	
23. Theoretical yield of $CaSO_4 \cdot xH_2O$	
24. Percent yield of $CaSO_4 \cdot xH_2O$	

CALCULATIONS

APPLICATION OF PRINCIPLES

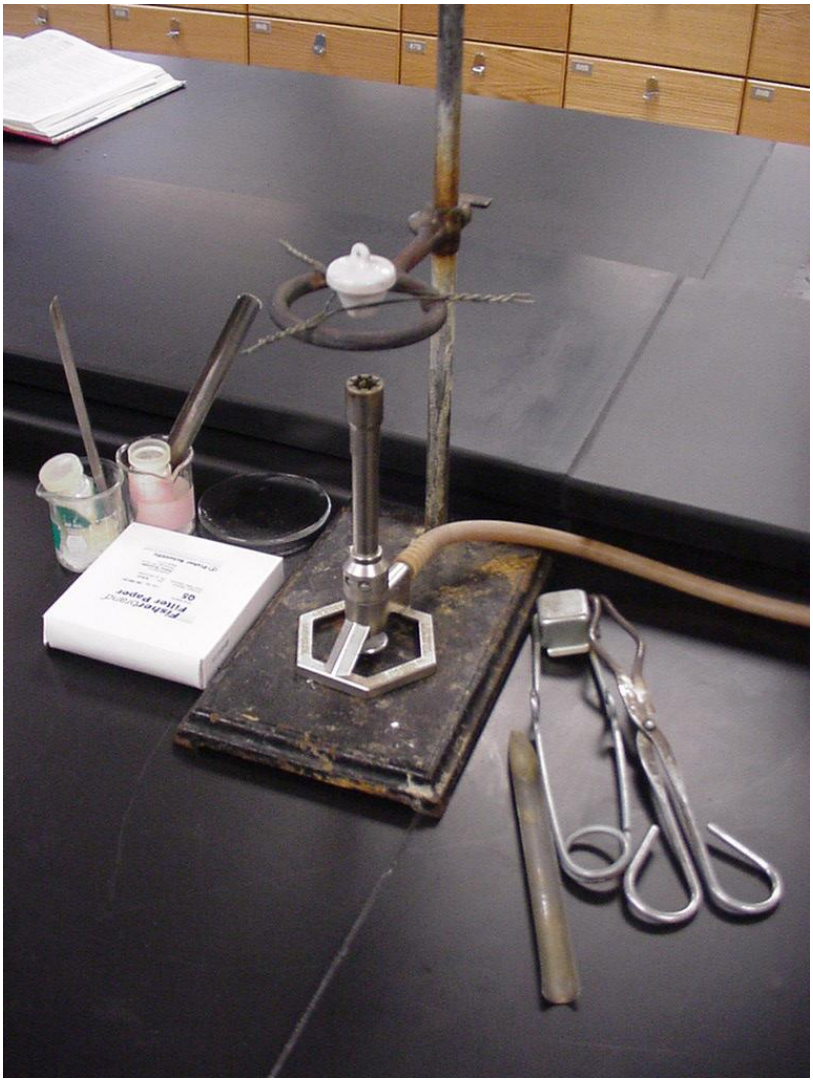
1. Suggest some reasons why the percent yield may not be 100%.

2. If 2.0 grams of anhydrous Na_2SO_4 are mixed with 2.0 grams of anhydrous $CaCl_2$, which chemical is the limiting reactant? Support your answer with a calculation.

3. If 2.0 grams of sodium sulfate decahydrate ($Na_2SO_4 \cdot 10H_2O$) are mixed with 2.0 grams of calcium chloride dihydrate ($CaCl_2 \cdot 2H_2O$), which chemical is the limiting reactant?

4. If 4.02 grams of a hydrate of magnesium sulfate loses 2.06 grams of water when heated, what is the formula for the hydrate?

5. If 2.0 grams of anhydrous sodium sulfate is mixed with 2.0 grams of calcium chloride dihydrate and 2.0 grams of calcium sulfate dihydrate are collected, what is the percent yield?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