EXPERIMENT 7

Copper Reactions

Objective

Seven reactions are carried out, and their qualitative features are carefully observed and recorded. The basic objectives of this investigation are to illustrate the following:

1. the four reaction categories;
2. some of the signs which indicate that a chemical reaction has occurred or is in progress;
3. the wide variety of stoichiometric ratios featured in chemical reactions;
4. transform copper into various copper compounds and reclaim pure copper.

Introduction

Several interesting concepts may be demonstrated by studying a sequence of reactions involving compounds of copper. Included in this series are double displacement, decomposition, acid-base, and oxidation-reduction processes.

A quantity of copper metal, issued to each student, is reacted in the first of several procedures to be given. The experiment will guide you in transforming copper into various copper compounds and back to pure copper.

In the final step, copper metal is recovered and weighed. A comparison of the mass of reclaimed copper with the mass of the original sample provides evidence of how carefully the experiment has been performed by calculating the percent recovery of the copper.

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

(EQ 7.1)

For each reaction, you will write a balanced chemical equation, calculate a theoretical yield, and classify the type of reaction. Example 7.1 below illustrates possible one transformation. It is not done is this experiment, but could be.

EXAMPLE 7.1

How would you prepare and isolate copper(II) sulfide from copper(II) nitrate?

Answer: You would mix it with a solution of potassium sulfide. The copper(II) sulfide would precipitate and you could filter the solution and the precipitate would contain pure copper sulfide. The potassium nitrate would remain in solution and be discarded with the filtrate.

Balanced chemical reaction:

$$Cu(NO_3)_{2\,(aq)} + K_2S_{\,(aq)} \rightarrow CuS_{\,(s)} + 2\ KNO_{3\,(aq)} \qquad \textbf{(EQ 7.2)}$$

Theoretical yield:

$$0.578\text{g Cu}(NO_3)_2 \times \frac{187.57\text{g Cu}(NO_3)_2}{65.38\text{mol Cu}(NO_3)_2} \times \frac{1\text{mol CuS}}{1\text{mol Cu}(NO_3)_2} \times \frac{95.61\text{g CuS}}{1\text{mol CuS}} = 0.294\text{ g CuS} \qquad \textbf{(EQ 7.3)}$$

Reaction Type: *Double displacement/precipitation*

Procedure

NOTE: You should have a clearly written log of what you did and saw. Be sure to note any unusual or unexpected observations in your running log. Good descriptions are important here.

Step 1: Preparation of copper(II) nitrate from copper metal

$$Cu_{\,(s)} + HNO_{3\,(aq)} \rightarrow Cu(NO_3)_{2\,(aq)} + NO_{2\,(g)} + H_2O_{\,(l)} \qquad \textbf{(EQ 7.4)}$$

1. Place the weighed sample of copper in a 250 mL beaker and slowly add 2 mL of 16 *M* $HNO_{3\,(aq)}$. The brown gas evolved is nitrogen dioxide, NO_2. Be sure to describe the appearance of your original piece of copper.
2. The reaction mixture may be heated on a hot plate to facilitate the reaction; do not boil the mixture to dryness.
3. Allow the mixture to stand until all the metal has reacted, and then slowly dilute the solution that remains with 20 mL of distilled water.

Step 2: Preparation of copper(II) hydroxide from copper(II) nitrate

$$Cu(NO_3)_{2\,(aq)} + NaOH_{\,(aq)} \rightarrow \qquad \textbf{(EQ 7.5)}$$

4. To the solution from "Step 1: Preparation of copper(II) nitrate from copper metal" on page 58, add 8 *M* $NaOH_{\,(aq)}$ solution drop by drop, with constant stirring. The solid that forms is copper(II) hydroxide.
5. Continue the addition of the 8 *M* $NaOH_{\,(aq)}$, frequently testing the solution with red litmus paper. This is readily done by touching a clean stirring rod to the solution and transferring a

drop of liquid to a piece of litmus paper. When the solution becomes basic, the litmus paper will turn blue.

6. At this point, discontinue the addition of $NaOH_{(aq)}$ since the precipitation of the hydroxide is essentially complete.

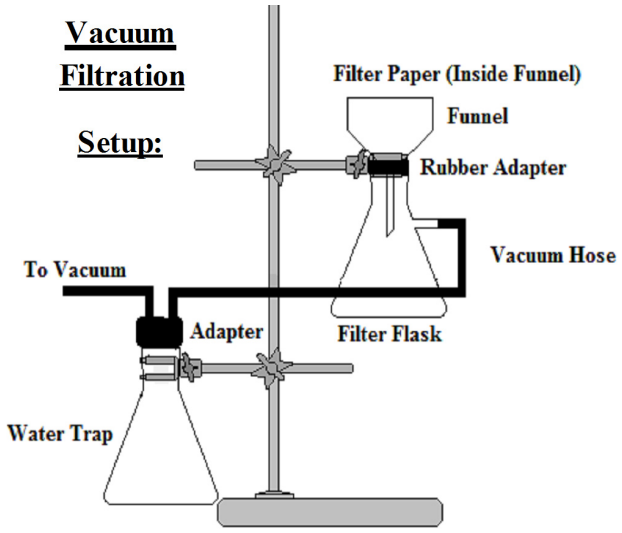
Be careful not to transfer any of the blue precipitate during this procedure since it can produce a blue coloration of the paper even though the solution is not alkaline.

Step 3: Preparation of copper(II) oxide from copper(II) hydroxide

$$Cu(OH)_{2\,(s)} \rightarrow \qquad \textbf{(EQ 7.6)}$$

7. Add 30 mL of deionized water to the mixture from "Step 2: Preparation of copper(II) hydroxide from copper(II) nitrate" on page 58.
8. Stirring constantly, gently boil the mixture until the solid copper(II) hydroxide is completely converted to copper(II) oxide.
9. The precipitate should settle to the bottom of the beaker as it boils. It may be stored until the next lab period at this point.
10. Perform a suction filtration and collect the copper(II) oxide in a Büchner funnel. A *fiberglass* pad filter paper should be used. Be sure to place the rough side up and wet it with water before beginning the filtration.

FIGURE 7.1



11. The filtrate may be discarded if it is free of black copper(II) oxide; otherwise it must be poured back into the funnel and filtered again.
12. Remove the suction from the filter flask and wash the solid in the funnel with a small volume of deionized water; apply suction in order to remove the wash liquid.
13. Repeat this operation and then discard all the wash solutions.
14. Do not store the solid collected here until the next lab period!

Step 4: Preparation of copper(II) sulfate from copper(II) oxide

$$CuO_{(s)} + H_2SO_{4\,(aq)} \rightarrow \quad \textbf{(EQ 7.7)}$$

15. Rinse the filter flask several times with distilled water. Reassemble the filtration apparatus, but do not apply suction during this step, a reaction with sulfuric acid is occurring.

16. Slowly add 2 *M* $H_2SO_{4\,(aq)}$ drop by drop to the funnel. Allow sufficient time for the solid to react in order to avoid using a large excess of the acid.

17. A total of 4 to 5 mL of the acid solution is required.

18. The contents of the funnel may be swirled, but this should be done carefully to avoid spillage.

19. There is a tendency for filter paper to lift off the surface of the funnel during this operation. This should cause no problem as long as any oxide which passes through the filter undergoes reaction in the filtration flask.

20. Continue the addition of 2 *M* $H_2SO_{4\,(aq)}$ until the oxide has completely reacted and all that remains is an aqueous solution containing copper(II) sulfate.

21. Apply suction to the filtration apparatus to draw the solution into the filter flask.

22. Rinse the funnel and filter paper with several small portions of deionized water and collect this wash liquid in the flask.

23. Carefully pour the contents of the filter flask into a clean 250 mL beaker.

24. Rinse the flask with a stream of deionized water, allowing the liquid to flow into the beaker.

Step 5: Preparation of copper(II) phosphate from copper(II) sulfate.

$$CuSO_{4\,(aq)} + Na_3PO_{4\,(aq)} \rightarrow \quad \textbf{(EQ 7.8)}$$

25. Add 8 *M* $NaOH_{(aq)}$ drop by drop, with constant stirring, to the solution from "Step 4: Preparation of copper(II) sulfate from copper(II) oxide" on page 60. This addition is continued until a blue precipitate of copper(II) hydroxide just begins to persist.

26. Then introduce 20 mL of 0.5 *M* $Na_3PO_{4\,(aq)}$ and stir the mixture thoroughly.

27. Allow any precipitate of copper(II) phosphate to settle and test the supernatant liquid with litmus paper.

28. If the liquid is acidic (indicator color is red), carry out a dropwise addition of 8 *M* $NaOH_{(aq)}$, with constant stirring, until it is just basic, and allow the precipitate to settle.

29. Carefully decant the supernatant, if possible, and dispose of it. Use an eye-dropper to remove as much of the remaining supernatant as possible without removing any precipitate.

30. Save all of the precipitate (note the color in your lab book) for"Step 6: Preparation of copper(II) chloride from copper(II) phosphate" on page 60.

Step 6: Preparation of copper(II) chloride from copper(II) phosphate

$$Cu_3(PO_4)_{2\,(s)} + HCl_{(aq)} \rightarrow \quad \textbf{(EQ 7.9)}$$

31. Add sufficient 6 *M* HCl with stirring to the beaker containing the copper(II) phosphate precipitate to dissolve all of the precipitate.

Step 7: Preparation of copper from copper(II) chloride

$$CuCl_{2\,(aq)} + Al_{(s)} \rightarrow \quad \textbf{(EQ 7.10)}$$

32. To the solution from "Step 6: Preparation of copper(II) chloride from copper(II) phosphate" on page 60 add a piece aluminum foil about the size of your filter paper and stir.
33. Two chemical reactions are evident: (a) the principal one in which a deposit of elemental copper appears; and (b) a side reaction in which hydrogen gas is evolved.
34. It may be necessary to introduce an additional small quantity of aluminum in order to complete the principal reaction.
35. Additional 6 *M* HCl also may be needed to react with any excess aluminum. The completion of the principal reaction is indicated by the disappearance of the blue color of the solution and the absence of further copper formation.

$$Al_{(s)} + HCl_{(aq)} \rightarrow \qquad \textbf{(EQ 7.11)}$$

36. To test for completeness of the reaction withdraw a 1-2 mL sample of the solution and add 6 *M* ammonia until you have a solution which is basic to litmus.
37. If copper is present you should observe the formation of a bright blue copper ammonium complex.
38. During the final stages of the reactions, weigh a piece of Whatman No. 4 filter paper. This paper should be suitable for gravity filtration of the copper metal product.
39. Proceed with the filtration once the formation of copper metal is complete and there are no pieces of aluminum remaining.
40. Fine particles of copper tend to pass through the filter paper, but usually the quantity is not significant and can be ignored.
41. Wash the copper with deionized water and then a few mL of acetone, a volatile liquid that permits the copper to dry rapidly.
42. Remove the filter paper from the funnel and spread it out on a dry watch glass so that the copper product may dry.
43. Meanwhile, weigh a clean, dry sample bottle. Be sure to describe the appearance of your reclaimed copper.
44. Place the dried, reclaimed copper and the filter paper in the sample bottle and reweigh.
45. Put a label on the bottle containing your name and the mass of the recovered copper.
46. Give the sample to your lab instructor for further evaluation.
47. The quality of the product depends on color and dryness as well as absence of visible impurities.

Calculations and Results

1. For each reaction you will:
    a. Write a description of what you did and what you observed.
    b. Write a balanced equation for the reaction and identify its type as double displacement, decomposition, acid-base, and/or redox.
    c. Calculate the theoretical yield (expected yield) of the product based upon the theoretical yield of the preceding step.

NOTE: You need to summarize your major data. This may be done several ways including: a list or a table

2. Obtain the mass of copper reclaimed in "Step 7: Preparation of copper from copper(II) chloride" on page 60.
3. Calculate the actual yield of the copper metal.
4. Calculate the percent recovery of the copper metal.
5. Calculate the percent error for the theoretical and actual amount of copper obtained.

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EXAMPLE 7.2

A 0.250 g sample of zinc metal was dissolved in 10.0 mL of 6.0 M HCl. When the shiny zinc was added to the HCl, it dissolved with the production of considerable gas to yield a colorless solution. The reaction was highly exothermic.

$$Zn_{(s)} + 2\ HCl_{(aq)} \rightarrow ZnCl_{2\,(aq)} + H_{2\,(g)} \qquad \textbf{(EQ 7.12)}$$

Reaction Type: Redox

$$\text{theoretical yield} = 0.250\,\text{g Zn} \times \frac{136.26\,\text{g ZnCl}_2}{65.38\,\text{g Zn}} = 0.521\ \text{g ZnCl}_2$$

If 0.4632 g of zinc chloride was recovered, what is the percent recovery?

$$\%\ \text{recovery} = \frac{\text{actual yield}}{\text{theoretical yield}} \times 100 = \frac{0.4632\,g}{0.521\,g} \times 100 = 88.9\%$$

Post Lab Questions

1. If you need to produce 3.50 kg of copper metal from a contaminated sample of reclaimed copper wiring, which is 68.3% copper, how many kg of reclaimed copper wiring would you need to buy if your percent yield at each step is 90%? Assume that you will isolate the copper wiring by going through the steps in this lab. Be sure to show all stoichiometry used.

2. How might you prepare (in one step) copper(II) nitrate from copper(II) chloride? Include in your answer: the reagent that you would add, a description of the equation that would take place, and an explanation of how you would then isolate the pure copper(II) nitrate from the reaction mixture.

3. How might you prepare (in one step) copper(II) hydroxide from copper(II) sulfate? Include in your answer: the reagent that you would add, a description of the equation that would take place, and an explanation of how you would then isolate the pure copper(II) hydroxide from the reaction mixture.