CHEMISTRY 205

Experiment 6A: ANALYSIS OF AN UNKNOWN OXIDE OF COPPER

Introduction

Copper has two common oxidation (or valence) states: copper (I) or *cuprous*, Cu^+ or Cu(I), and copper(II) or *cupric*, Cu^{2+} or Cu(II). Because of this, it is possible to obtain two oxides of copper: cuprous oxide, Cu_2O, and cupric oxide, CuO. In this experiment, you will be given one of these two oxides, and your task is to determine which one you have.

The method you will use in this experiment falls into the general class of techniques called *gravimetric analysis*. Gravimetric analysis is a method of analysis that uses the mass of product formed during a reaction as a means to investigate the nature of one of the reactants. There are typically two contexts in which gravimetric analysis is used: either (1) you know the identity of the compound but not how much of it is present in your sample, *or* (2) you know how much of it you have, but not exactly what it is! In the second case, if you happen to know that there are only a couple of potential identities for your compound, then you can likely find a simple reaction or two that would occur for both possibilities. If so, by analyzing the quantities of products and thinking about the stoichiometry of the reactions, you can determine which reactant you originally had.

In this experiment, the analytical method is based on precipitating metallic copper from a known mass of an unidentified oxide of copper. The equations for the reactions involved are shown below in the Procedure Summary. By isolating and weighing the precipitated Cu(s), the amount of oxygen that was combined with the copper in the original compound can be determined by difference. Using this information, the elemental composition of the oxide can be calculated, which will ultimately establish the identity of the oxide.

Procedure Summary

In this experiment, the unknown copper oxide (which is insoluble in water) will first be dissolved in hydrochloric acid to give a solution of copper chloride. Then the solution will be treated with metallic aluminum; this will result in the formation of aluminum chloride and the precipitation of copper metal. The two possible reaction sequences are:

$$Cu_2O(s) + 2HCl(aq) \rightarrow 2CuCl(aq) + H_2O(l)$$
$$3CuCl(aq) + Al(s) \rightarrow AlCl_3(aq) + 3Cu(s)$$

or

$$CuO(s) + 2HCl(aq) \rightarrow CuCl_2(aq) + H_2O(l)$$
$$3CuCl_2(aq) + 2Al(s) \rightarrow 2AlCl_3(aq) + 3Cu(s)$$

The reactions with aluminum are examples of oxidation - reduction (redox) reactions, and involve the following half reactions:

$$Al \rightarrow Al^{3+} + 3e^-$$

and

either $Cu^+ + e^- \rightarrow Cu$

or $Cu^{2+} + 2e^- \rightarrow Cu$

Both copper(I) and copper(II) have a stronger tendency to gain electrons than aluminium(III); in other words, aluminum ion is more electropositive than copper ions (which are more electronegative). For this reason, electrons will be passed from the aluminum metal to the copper ion according to one of the complete reactions shown above.

Prelaboratory Assignment

Read the procedure and answer the prelaboratory questions before coming to the lab. Your demonstrator will inspect and collect in your prelab before you are permitted to begin the experiment - keep a copy of it for yourself, and have the TA sign your receipt record.

Materials

Apparatus

- 100 mL beaker
- medicine dropper
- glass rod
- hot plate at low heat

Reagents and Materials

- copper(I) or copper(II) oxide
- 6M hydrochloric acid **[Caution – corrosive]**
- distilled water
- about 0.1g aluminium metal (strip)
- acetone

Procedure

NOTE: You will be working with small quantities of quite concentrated hydrochloric acid. Be careful not to get any on your skin, eyes or clothing. Work in a fume hood to avoid inhaling hydrogen chloride gas, the white fumes that diffuse out of concentrated hydrochloric acid solutions. Wipe up small spills immediately - consult your demonstrator in the case of a serious spill.

1. Weigh a clean dry 100 mL beaker (to the nearest milligram). In the beaker, weigh out between 0.15 and 0.20 g of the copper oxide that you are given. Report the weights.
2. Using a medicine dropper, carefully add about 2.5 mL of 6M hydrochloric acid. Swirl the contents of the beaker to make sure the copper oxide is thoroughly wetted by the acid. The oxide will gradually dissolve over a few minutes. Swirl the mixture occasionally and warm it at *low* heat on a hot plate.
3. When the copper oxide has completely dissolved, dilute the solution with about 2.5 mL of distilled water. Then add the aluminum. Report what happens, and specifically, how you determine that the reaction is complete.
4. When all the copper has been precipitated, fish out the remaining aluminum with a glass rod (avoid removing any of the copper with it) and place it in the waste container provided. Carefully decant off as much of the liquid in the beaker as you can with a pipette *without removing any of the copper*. (The acidic aluminum chloride solution can be flushed down the sink.) Add about 5 ml of deionized or distilled water and decant again to wash the last traces of aluminum chloride solution off the copper; do this twice.
5. Wash the wet copper twice with about 2 mL of acetone, again by decantation. This will dissolve the water in the acetone, and leave the copper wet with the more volatile acetone (boiling point = 56 °C). The remaining acetone is evaporated by heating the beaker *gently* on the hot plate.
6. Cool the beaker to room temperature and weigh it to determine the mass of copper obtained.

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Experiment 6A: ANALYSIS OF AN UNKNOWN OXIDE OF COPPER

Laboratory Report

Observations and Data

1. (2 marks) Describe what you observed upon addition of the hydrochloric acid to the copper oxide.

   It changed to white ~~[illegible]~~ and then black

2. (2 marks) Describe what you observed upon addition of the aluminum to the copper-containing solution.

   bubbels gas released, voilent reaction Aluminum became ~~[illegible]~~ black then presipitate + copper has formed.

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3. (2 marks)

Mass of beaker (g)	49.452 g
Mass of beaker plus copper oxide (g)	49.618 g
Mass of copper oxide (g)	0.166 g
Mass of beaker plus copper (g)	49.593 g
Mass of copper obtained (g)	0.141 g
Mass of oxygen in the copper oxide (g)	0.025 g

Conclusions and Interpretation of Results

4. (1 mark) Which oxide do you think you were given based solely on your answers to the prelab questions? Explain.

5. (2 marks) Calculate the observed percentage composition of your copper oxide. Show your calculations. From this result, deduce which pure oxide you were given.

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6. (2 marks) Do the observations you have reported, and the properties you looked up for the prelab questions, support your calculated result? Explain.

Additional questions

7. (2 marks) Calculate how many millilitres of 6M hydrochloric acid were actually required to react with your sample of copper oxide (show your calculations). Approximately what percentage excess did you use?

8. (2 marks) Calculate the *observed* empirical formula of your copper oxide, *i.e.*, the formula based exactly on your experimental results (note: you will likely have fractional stoichiometric subscripts here).

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Experiment 6B: GRAVIMETRIC DETERMINATION OF CARBONATE OR BICARBONATE

Introduction

The premise of this experiment is the same as Experiment 6A, in which you used gravimetric analysis to identify an unknown oxide of copper. In this experiment, you will identify an unknown as either sodium carbonate ("soda") or sodium hydrogen carbonate (sodium bicarbonate or "baking soda"). The method will involve the reaction of the unknown with excess hydrochloric acid, which will occur according to one of the two following reactions:

$$Na_2CO_3(aq) + 2HCl(aq) \longrightarrow 2NaCl(aq) + CO_2(g) + H_2O(l)$$

$$NaHCO_3(aq) + HCl(aq) \longrightarrow NaCl(aq) + CO_2(g) + H_2O(l)$$

Both reactions produce the same products, but not in the same proportions. Thus, gravimetric analysis of the solid product, NaCl, can be used to determine the identity of the unknown.

Procedure Summary

A known mass of the unknown compound will be reacted with excess hydrochloric acid. The products of the reaction are carbon dioxide, sodium chloride and hydrogen chloride; these compounds and the solvent in which they will be dissolved (water) are all volatile species, except for sodium chloride. Thus, even though all the products are soluble in water, heating the solution containing them will drive off everything except the sodium chloride. After evaporating the product mixture to dryness, the sodium chloride residue will be weighed. Comparison of the mass of the starting material with the mass of sodium chloride obtained will allow for the identification of the unknown based on the reaction's observed stoichiometry.

Pre-Laboratory Assignment

Read the procedure and answer the pre-laboratory questions before coming to the lab. Your demonstrator will inspect and collect in your prelab before you are permitted to begin the experiment - keep a copy of it for yourself, and have the TA sign your receipt record.

Materials

Apparatus

- 100 mL beaker
- hot plate (on high)
- medicine dropper

Reagents and Materials

- unknown (anhydrous sodium carbonate *or* sodium hydrogen carbonate)
- 6M hydrochloric acid

Procedure

You will be working with small quantities of quite concentrated hydrochloric acid. Be careful not to get any on your skin, eyes or clothing. Avoid inhaling the vapour - work in a fume-hood. Wipe up small spills immediately - consult your demonstrator in the case of a serious spill.

1. Weigh the beaker to the nearest milligram and report its mass. Place about 0.2 g of the unknown substance in the beaker. Then weigh the beaker again. Report the mass.
2. Add two drops of the 6M hydrochloric acid to the beaker. Note what happens. Continue adding hydrochloric acid dropwise until there is no more reaction, and then add a couple more drops to be absolutely sure that an excess of acid has been added.
3. Heat the beaker on a hot plate so that the solution boils; evaporate the mixture to dryness. Be careful near the end so that the contents of the beaker do not splatter excessively (to prevent loss of product). Continue heating the beaker to ensure it and its contents are truly dry. (Any water/hydrochloric acid that condenses near the top of the beaker can be blotted up with a piece of paper towel - wear gloves! Take care not to remove any sodium chloride solution; *i.e.*, only wipe off condensate, not splattered solution.)
4. Allow the beaker to cool to room temperature and then weigh it again to determine the mass of the sodium chloride residue. The sodium chloride can be then redissolved in water and flushed down the sink.

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Experiment 6B: GRAVIMETRIC DETERMINATION OF CARBONATE OR BICARBONATE

Laboratory Report

Observations and Data

1. (5 marks) (Report masses in grams.)

Mass of empty beaker	67.310g
Mass of beaker and unknown	67.506g
Mass of unknown	.197 g
Moles of unknown assuming it is sodium carbonate	1.86×10^{-3}
Moles of unknown assuming it is sodium hydrogen carbonate	2.34×10^{-3}
Mass of beaker and sodium chloride	67.460g
Mass of sodium chloride	.15 g
Moles of sodium chloride	2.56×10^{-3}

2. (3 marks) Calculate the predicted yield (in g) of NaCl assuming your unknown is sodium carbonate.

[illegible]
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3. (3 marks) Calculate the predicted yield of NaCl (in g) assuming your unknown is sodium hydrogen carbonate.

4. (3 marks) What is the most probable identity of your unknown? Explain.

Additional Question

5. (1 mark) Suppose your unknown had been a hydrated form of sodium hydrogen carbonate with the formula $NaHCO_3 \cdot H_2O$. What would differ in your calculations? Could you still have distinguished it from sodium carbonate? Explain.