

The Copper Cycle – Compounds of Copper

INTRODUCTION:

Properties of copper:

Copper is a **transition metal element** which is usually found in nature chemically bonded to various other elements. Some of the most commonly found forms of copper are the sulphides of copper such as covellite (CuS, which is also called cupric sulphide or copper(II) sulphide), chalcocite (Cu₂S, which is also called cuprous sulphide or copper (I) sulphide), and chalcopyrite (CuFeS₂).

Copper is a fairly soft and malleable metal with a characteristic bright orange-brown colour which is often called the “copper colour”. Copper metal is an excellent **conductor** of both **heat** and **electricity** and mix well with other metals to give many kinds of **alloys** such as **bronze** (copper mixed with tin) and **brass** (copper mixed with zinc). Alloys of copper are much harder and stronger than metallic copper. Since copper metal is soft, mixing copper with other metals to produce alloys is necessary for practical purposes.

Copper does not oxidize(lose electrons) readily in air and has a very low reactivity with dilute mineral acids such as sulfuric acid (H₂SO₄) and hydrochloric acid (HCl). However nitric acid (HNO₃) has a high reactivity with copper and will readily dissolve copper metal producing the copper(II) ion. Dissolving of copper metal in nitric acid is an oxidative process where electrons are transferred from copper metal to the nitrogen of the acid.

Centrifuging:

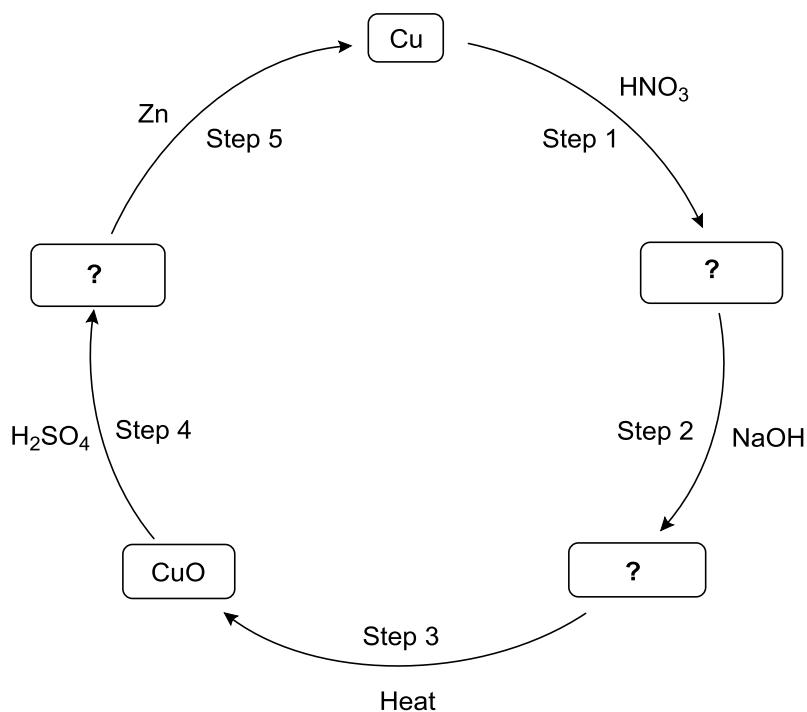
In some chemical reactions when two solutions are mixed a solid product called the **precipitate** will form. Once the precipitate has been formed, the liquid part above the precipitate is called the **supernatant** and often particles of the precipitate will be floating in the supernatant. In order to obtain good separation between the precipitate and the supernatant the reaction mixture has to be **centrifuged**. Centrifuging is the process of separating solid and liquid components of the mixture by spinning a sample around a fixed axis. Once the centrifuged the

supernatant can be carefully poured out leaving the precipitate behind in a process called **decantation**.

OBJECTIVE:

In this experiment copper metal will be treated successively with nitric acid, sodium hydroxide, heat, and sulfuric acid to convert the copper metal into a series of compounds of copper. Finally using metallic zinc the copper metal will be regenerated and the percent recovery of copper metal will be determined.

The five step process of the copper cycle is summarized in the following diagram. [The regenerated copper metal will be recovered in the last and the sixth step of the experiment.]



PROCEDURE:

CAUTION: EXPERIMENT USES CORROSIVE NITRIC AND SULFURIC ACID REAGENTS!!

ALL WASTE IN THIS EXPERIMENT CAN BE FLUSHED DOWN THE SINK WITH LOTS OF WATER.

Step 1: Dissolution and Oxidation

Obtain a clean dry test tube and **weigh** it on the analytical balance. Add around 0.1 g of copper metal to the test tube and record the exact mass of the test-tube with copper (to 4 decimal places) using the analytical balance. Calculate the mass of copper metal used. Using a glass stirring rod push the copper metal to the bottom of the test tube. Place the test tube containing copper metal in the fume hood. **CAUTION: Nitric acid is corrosive and the reaction is vigorous.** Add 1 mL of concentrated nitric acid. Once the reaction is complete (when all the copper is dissolved and no more evolution of fumes) add 1 mL of distilled water. When copper metal reacts with nitric acid, three products are formed and include the aqueous copper ions (in the presence of excess NO_3^- ions from the nitric acid), $\text{NO}_2(\text{g})$ and $\text{H}_2\text{O}(\text{l})$. Record the observations of this step in your data sheet.

Step 2: Conversion

Add 5 mL of 6 M NaOH solution to the test tube containing the reaction mixture from step 1. Mix the contents of the test tube with a glass stir rod and centrifuge the test tube to obtain good separation between the precipitate and the supernatant. Record the observations of this step in your data sheet. The copper compound from step 1 undergoes a **double displacement reaction** in this step.

Step 3: Decomposition

Centrifuge your test tube as demonstrated by your lab instructor and carefully decant the supernatant from the centrifuged test tube. Using a test tube holder, place the test tube in a hot water bath until the precipitate turns black. The black precipitate is copper(II) oxide (CuO). The

copper compound from step 2 undergoes decomposition. The production of CuO through the decomposition reaction is quite fast and it is not necessary to wait until the product of step 2 to be completely dry. Record the observations of this step in your data sheet.

Step 4: Soluble Salt

To the CuO obtained in step 3, add about 5 mL of 6 M H₂SO₄ (or enough to dissolve all of the CuO) and carefully shake the test tube to dissolve the solid. The sulfuric acid along with CuO will form a water soluble salt of copper through a **double displacement reaction**. Record the observations of this step in your data sheet.

Step 5: Regeneration of Copper Metal

Using a scoopula add a small piece of zinc wire or ribbon into the test tube. If necessary wash the inside walls of the test tube with small amounts of distilled water. Periodically agitate the test tube using a glass stirring rod making sure to carefully grind the metal pieces. Place the test tube in a hot water bath and repeat the grinding, mixing and heating until the solution in the test tube turns colourless. If the solution is still blue and there is no sign of reaction add more zinc. Record your observations of this step in your data sheet.

Step 6: Removal of Excess Zinc Metal and Recovery of Copper Metal

Once the reaction mixture in the test tube of step 5 has turned completely colourless, carefully decant and discard the supernatant of the test tube as much as possible without losing any of the solid, which is unreacted zinc and newly formed copper metal. Add 1-2 mL of 6 M HCl to the solid product in the test tube. The HCl will react and oxidize the excess zinc generating zinc(II) ions and hydrogen(H₂(g)) and leaving the solid copper metal unreacted. Leave the test tube for 5 – 10 minutes and stir occasionally to assure completion of reaction. Add another 1 mL of 6 M HCl to check the completion of reaction. If the evolution of gas bubbles has not stopped continue adding 6 M HCl and continue stirring until the evolution of gas bubbles stop.

Once all the zinc has been dissolved, decant the supernatant of the test tube and wash the contents 3 times with 3 mL portions of distilled water. This is done by adding and decanting water from the test tube. Repeat the washing process of the copper metal with acetone. Place the

test tube in an oven for 10 minutes for further drying. Once completely dry cool the test tube to room temperature and obtained the combined mass of the test tube and the recovered copper metal. Record your data in the datasheet.

As a control experiment place a piece of copper in a separate dry test tube and add 2 mL of 6 M HCl. Record your observations for both test tubes in your data sheet.

Quiz Hints:

1. Know the properties of copper metal.
2. Know the oxidation state changes and how to determine oxidation state.
3. Know how to write balanced chemical reactions.
4. Know definitions and terms discussed in the lab handout.
5. Know the observations and colour changes of each step in the copper cycle.
6. Know how to do a percent recovery calculation.
7. Know possible experimental errors.