

Chemistry 1215 Experiment #9
Copper and its Compounds

Objective

The objective of this experiment is to take a piece of copper as efficiently as possible through a series of chemical reactions. The final reaction in the sequence ultimately produces copper metal. Each chemical reaction will be classified and described using a balanced chemical equation.

Introduction

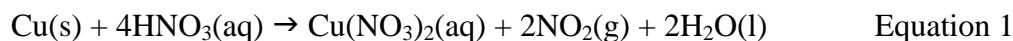
Oxidation-reduction reactions (sometimes called redox reactions) can be identified because the oxidation numbers of some of the elements on the reactant side of the chemical equation are different than their oxidation numbers on the product side of the equation. See your lecture text for a discussion on assigning oxidation numbers. The oxidizing agent in an oxidation-reduction reaction is the reactant that gains electrons, that is, it is reduced. The reducing agent is the reactant that loses electrons and is oxidized. In other words, the reducing agent acts to reduce something else and is oxidized in the process.

Copper is a member of the first row transition metal series. It has physical properties typically associated with metals. It is an excellent conductor of heat and electricity and is used extensively in electrical wiring and in pots and pans. Because of its distinctive color it has been used for thousands of years for decorative purposes. One quarter of the copper produced in the United States each year comes from the massive open pit mine owned and operated by Kennecott in Utah. The amount of copper in the copper containing ore is only 0.6%. Huge amounts of rock must be removed from the pit and crushed before the copper can be extracted.

Copper is relatively inert chemically, that is it is not very reactive. However, it does react readily with nitric acid. This reaction is the starting point for today's reaction.

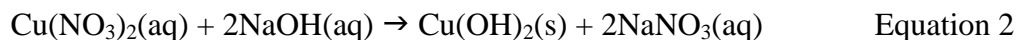
Reaction 1: Copper and Nitric Acid

Copper metal is not generally soluble in acid because copper is a stronger reducing agent than hydrogen, that is, copper metal will not reduce H^+ to H_2 . Because copper is not oxidized by H^+ , it is inert to most acids. Aqueous nitric acid, $HNO_3(aq)$, however, is able to dissolve copper metal because nitrate, NO_3^- , is a powerful oxidizing agent. In nitric acid, copper metal is oxidized, that is it loses two electrons to form Cu^{+2} ions which then are soluble in the water solvent giving the solution a distinctive blue color. Whenever an oxidation occurs there must also be a corresponding reduction (see equation 1), that is the electrons lost by copper must be passed to another atom or atoms. In this case the nitrogen of nitric acid is reduced from NO_3^- , with an oxidation number of +6, to $NO_2(g)$, which has an oxidation number of +4, a gain of two electrons. Nitrogen dioxide is a noxious brown gas that will readily react with biological tissues such as skin, lungs, etc. and therefore should be avoided. Consequently, it is imperative that this step of this experiment be carried out in a working fume hood. Once copper is oxidized to copper(II) ions it forms soluble, aqueous copper (II) nitrate (see Equation 1).



Reaction 2: Copper Nitrate and Sodium Hydroxide

The blue copper nitrate solution is acidic due to the presence of excess nitric acid used in the first step. Sodium hydroxide, a strong base, is next added to neutralize the acid. Addition of excess sodium hydroxide solution allows a double displacement reaction, also called a precipitation or metathesis reaction, to occur as the copper (II) ions react with hydroxide ions to produce insoluble, deep blue copper (II) hydroxide (see Equation 2). The other product of this reaction, sodium nitrate, is soluble in water and thus stays in solution.



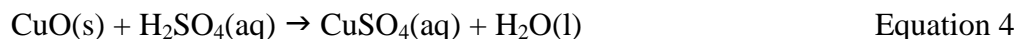
Reaction 3: Decomposition of Copper (II) Hydroxide

The blue copper(II) hydroxide decomposes upon heating to form black copper(II) oxide and water vapor (see Equation 3). Note that the copper is still in the form of Cu^{+2} , that is, it has not undergone oxidation or reduction.



Reaction 4: Copper Oxide and Sulfuric Acid

Copper(II) oxide will dissolve in dilute sulfuric acid to form an aqueous blue solution containing Cu^{+2} and sulfate ions as described by equation 4.

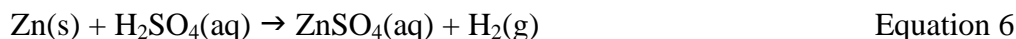


Reaction 5: Copper(II) Sulfate and Zinc Metal

The addition of zinc metal to the copper(II) sulfate solution formed in reaction 4 results in a replacement reaction in which zinc metal is oxidized to zinc ions and the copper(II) ions in the solution are reduced to copper metal (see Equation 5).



In order to determine the mass of copper metal produced in reaction 5, it is essential that there be no zinc metal present. One way to do this is to be scrupulously careful in adding the zinc to ensure no extra zinc is added. This solution is not practical in the amount of time we have in the lab although it is still to your advantage to add the least amount of zinc possible. Any excess zinc metal can then be removed by adding dilute sulfuric acid producing soluble zinc sulfate and hydrogen gas (see equation 6). Remember that copper metal is insoluble in acid.



Procedure

Reaction 1: Dissolving the Copper

1. Obtain a clean, dry, glass centrifuge tube.

2. Place a piece of copper wire in a weighing paper, determine the mass of the wire and place it in the centrifuge tube. The copper wire should weigh less than 0.0200 grams.
3. **In a fume hood**, add seven drops of concentrated nitric acid to the reaction tube so that the copper metal dissolves completely. Describe your observations in the lab report. (Caution, Concentrated nitric acid and nitrogen dioxide are very corrosive. Either will turn your skin yellow on contact. Do not leave any spills on the lab bench or in the fume hood.)
4. When the copper has dissolved, add seven drops of distilled water to the tube.

Reaction 2: Preparation of Copper(II) Hydroxide

1. Add 15 drops of 3.0 M aqueous sodium hydroxide to the tube. Make sure that the reactants are well mixed. Shake the tube carefully or gently flick the bottom of the tube with your finger. Remember that the contents of the tube may still be corrosive.
2. Add a second 15 drops of NaOH(aq), mix well, and record your observations. If you have two layers at this point it means that you have not mixed the solution well enough.
3. Centrifuge the reaction mixture.
4. The liquid at the top of the centrifuged mixture is called the supernatant while the solid is called a precipitate. Before separating the supernatant from the precipitate it is necessary to ensure that all of the copper(II) hydroxide has been precipitated. The supernatant should be clear and colorless indicating the absence of any Cu^{+2} ions in the solution. It should also be basic due to an excess of OH^- ions. Using a clean glass stirring rod, transfer a drop of the supernatant onto a piece of red litmus paper. If the litmus paper turns blue then the solution is basic and enough NaOH has been added. If the paper does not turn blue, add more NaOH, mix well, recentrifuge, and repeat the litmus paper test until the paper does turn blue.
5. An efficient separation of supernatants and precipitates is key to obtaining a good final yield of copper. The supernatant liquid can be separated from the precipitate by expelling the air from the bulb of a Pasteur pipet, inserting the tip of the pipet into the supernatant, then gently sucking the supernatant into the pipet. If you expel air or liquid into the precipitate with the pipet, you will stir up the precipitate and will have to repeat the centrifugation step. Remove as much liquid as possible and discard it in the waste container provided on the instructor's cart. It is better to leave a small amount of supernatant liquid than to remove some of the copper(II) hydroxide precipitate.

Reaction 3: Formation of Copper(II) Oxide

1. Set up a hot water bath by placing a beaker of water on a hotplate, placing an iron ring around the beaker, and heating the water to boiling.
2. Place the centrifuge tube containing the copper(II) hydroxide into the boiling water. Carefully hold the tube with a test tube clamp so that it doesn't get water into it. Record your observations.

Reaction 4: Formation of Copper(II) Sulfate

1. Add 20 drops of 3.0 M H_2SO_4 to the solid in the centrifuge tube. Stir carefully to ensure that the copper(II) oxide dissolves completely. Complete dissolution of the mixture will require thorough mixing and possibly heating of the solution.
2. Obtain the mass of a small, clean, glass test tube as accurately as possible.
3. Transfer the liquid from the centrifuge tube into the test tube. Rinse the centrifuge carefully with 1.0 mL of distilled water and transfer the rinse into the test tube containing your sample.
4. Record your observations on the data sheet.

Reaction 5: Formation of Copper Metal

1. Add a small quantity of zinc powder to the sample solution. Continue adding zinc in small quantities until the solution loses the blue copper(II) color. Any excess zinc added will need to be removed so don't add it too quickly or in large quantities. When the solution has turned colorless, add several drops of 3.0 M H_2SO_4 to the tube to dissolve any left over zinc. You can tell that the zinc has dissolved when addition of sulfuric acid does not generate bubbles.
2. Allow the copper metal to sink to the bottom of the tube and carefully remove the supernatant liquid using a Pasteur pipet.
3. Wash the red-brown copper metal in the tube with 1.0 mL of water. Allow the copper metal to settle to the bottom and remove the excess water. Repeat this rinsing process two more times.
4. Describe your observations on the data sheet.

Drying the copper metal

1. After removing as much of the third rinse water as possible you are ready to dry the metal. This must be done carefully in a cool Bunsen Burner flame. If the tube is heated too quickly there is a risk of ejecting the contents of the tube as the water boils. Also, if the flame is too hot you may convert the copper metal back into black copper(II) oxide. The objective is to drive the water from the tube as steam. Make sure that as water condenses on the walls of the tube that you continue to heat until all of the water is gone from the tube.
2. Once all of the water is removed from the tube, cool the tube and its contents then determine the mass of copper by weighing the tube and subtracting the tube + copper weight from the weight of the empty tube (Reaction 4 step 2). If the mass of copper is higher than the original mass of the copper wire it either contains water or zinc or has been converted to copper(II) oxide. Excess water can be removed by reheating the tube and reweighing to constant mass. Excess zinc requires addition of sulfuric acid followed by re-rinsing with water and re-drying.

Chemistry 1215 Experiment 9 Lab Report
Name _____

Data Sheet

Mass of copper wire _____

Mass of clean, dry test tube _____

Mass of test tube plus copper _____

Mass of final copper sample _____

Percent recovery of copper. Show all calculations.

Observations

1. Describe your observations for Reaction 1 including colors, gases formed, etc.
2. Describe your observations for Reaction 2 including colors, gases formed, etc.
3. Describe your observations for Reaction 3 including colors, gases formed, etc. Estimate the temperature of the decomposition of Copper(II) hydroxide.
4. Describe your observations for Reaction 4 including colors, gases formed, etc.
5. Describe your observations for Reaction 5 including colors, gases formed, etc.

Write a brief discussion of your results including a statement of the final percent recovery of copper and a discussion of reasons why the recovery differs from 100%.

2. Copper metal doesn't "rust" in the presence of oxygen at room temperature. However, it will react with O_2 at elevated temperatures. Write a balanced chemical equation describing the formation of copper (II) oxide when copper metal is heated in air.
3. When zinc is dissolved in sulfuric acid a gas is produced. What is the chemical identity of this gas? How is it produced?
4. Jaffrey Zagnut started with a 0.032 g sample of copper which he took through the series of reactions described in this experiment. At the end of the experiment he obtained 0.038 g of a black product. What was his percent yield? What is the most likely source of the error in his experiment? (Hint: consider question 2 above)