CHEM 231

Experiment 3

A Cycle of Copper Reactions

In this experiment, you will begin with the element copper, and carry out a series of chemical transformations in which you will see copper in other forms. The last reaction returns the copper to its original metallic form which you will recover and weigh. The purposes of the experiment are

- to observe various types of chemical reactions and relate observations to these reactions.
- to reinforce the idea of atoms being preserved in chemical reactions.
- to practice quantitative techniques by attempting to recover the original copper and computing the percent recovered.

Please note that in their pure forms, all of the compounds observed are solids. In this experiment, we may not see them as solids because they may be dissolved in water. Water is present from the first step until the recovered copper is dried in the last step. In making observations, it is important to distinguish between a solution and a solid suspended in water.

Procedure

Step 1: The oxidation of copper metal by nitric acid

Cut a length of pure copper wire that weighs about 0.5 g (about a 10-cm length). Polish it with a piece of sandpaper to remove any oxides or other coating. (Some copper wire may have a very thin plastic coating.) Weigh the wire to at least to the nearest mg and record this mass. Coil the wire and place it in the bottom of a 250-mL beaker.

The next step must be done in a fume hood because of the possibility of noxious gas being produced. Add 4.0 mL of concentrated nitric acid (a solution of HNO_3). Swirl the solution around in the beaker until the copper has completely dissolved. Add water until the beaker is about half full. The remainder of the experiment can be carried out at your lab bench. Record in your notebook a description of everything you see in this step.

Background information

The reaction that takes place can be written two different ways. Here is the first:

$3Cu(s) + 8HNO_3(aq) \rightarrow 3Cu(NO_3)_2(aq) + 2NO(g) + 4H_2O$

Another way of writing the reaction recognizes the fact that ionic compounds in solution exist as separate ions when they are in solution and the same is true of strong acids such as nitric acid.

$3Cu(s) + 8H^+(aq) + 8NO_3^-(aq) \rightarrow 3Cu^{2+}(aq) + 6NO_3^-(aq) + 2NO(g) + 4H_2O$

The nitric oxide gas (NO) that is produced is not observed in this experiment because it almost instantly reacts with atmospheric oxygen to produce nitrogen dioxide: $2NO(g) + O_2(g) \rightarrow 2NO_2(g)$

*The reaction involving copper is an example of an oxidation reaction. When an element increases in charge it is said to be **oxidized**.*

In solution, most ions are colorless. Copper(II) ion, however is an exception.

Questions

Based on the background information and your observations, answer the following questions before continuing:

What element is oxidized?

What observations can you make about nitrogen dioxide gas?

Where is the original copper after the reaction?

Is copper(II) nitrate soluble or insoluble?

The above reactions should be written in your notebook and all observations should be related to the compounds which appear in those reactions.

Step 2: Formation of copper(II) hydroxide

While stirring the solution from the last step with a glass rod, add 30 mL of 3.0 M NaOH. Write down any observations you make directly in your notebook.

Background information

Sodium hydroxide is a strong base which can neutralize acids. The solution you have from the first step was made with excess nitric acid and thus is strongly acidic. After all the excess acid is gone, additional sodium hydroxide can react with the copper compound in the following manner:

$Cu(NO_3)_2(aq) + 2NaOH(aq) \rightarrow Cu(OH)_2(?) + 2NaNO_3(aq)$

Questions

Answer the following questions before continuing:

Is copper(II) hydroxide soluble or insoluble? How can you tell? In other words, should the phase designation in the above reaction be (aq) or (s)?

What color is copper(II) hydroxide?

The reaction should be written in your notebook and any observations related to the reactants and products of that reaction.

Step 3: Formation of copper(II) oxide

Stirring gently with a glass rod to prevent "bumping," heat the solution just barely to the boiling point over a Bunsen burner. You should see a distinct change in the appearance of the suspended material. Record the observed changes in your notebook. When the transformation is complete, remove the beaker from the flame and continue stirring for a minute or so. Then allow the suspended material to settle. Decant (pour off) the liquid on top. Be careful not to lose any of the settled solid. Add about 200 mL of distilled water, allow to settle again, and decant again. Repeat this process two more times.

Background information

The hydroxide ion is OH^- and the oxide ion is O^{2-}. Ionic compounds made from these ions are often closely related. If you consider the formula of copper(II) hydroxide and remove from it one O atom and two H atoms (one water molecule), you will see that what remains is the formula for copper(II) oxide. Copper(II) hydroxide can, therefore, be called a "hydrate" of copper(II) oxide. In this case, heating the hydroxide is able to drive off the water from the solid, creating the oxide. This is accomplished even though the entire process is carried out in the presence of water. The purpose of the final rinsing and decanting process is to remove excess sodium, nitrate and hydroxide ions from the mixture, leaving you with only a suspension of the solid in water.

Questions

Answer the following questions before continuing:

What is the formula of copper(II) oxide?

What color is copper(II) oxide?

Is it soluble or insoluble?

Write a balanced equation showing the conversion of copper(II) hydroxide to copper(II) oxide.

Step 4: Formation of copper(II) sulfate

Add 15 mL of 6.0 M H_2SO_4, while stirring. Record your observations.

Background information

*Sulfuric acid is a strong acid and in the right circumstances can give up two H^+ ions. When it does, the sulfate ion remains. In this step the hydrogen ions are taken up by the oxide ion, producing water as a product. The net overall reaction is $CuO + H_2SO_4 \rightarrow CuSO_4 + H_2O$. This reaction is an example of an **acid/base** reaction. In such a reaction some compound gives up H^+ ions to another compound. The substance giving up the H^+ ions is called the **acid** and substance accepting them is called the **base**.*

Questions

Answer the following questions before continuing:

What is the acid and what is the base in this reaction?

Is the copper(II) sulfate product soluble or insoluble?

What color is copper(II) sulfate?

Did the copper change its charge in this reaction?

Rewrite the chemical equation for this process including phase designations.

Step 5: Formation of metallic copper

In the fume hood, add 2.0 g of powdered zinc metal, stirring until any reaction is apparently complete. Record n your observations.

Background information

*In the first step of the reaction, copper metal was oxidized from 0 charge to the +2 state. In the subsequent reactions, the copper atoms remained in the +2 state. To recover the copper metal, the copper ions must be returned to their native state of 0 charge. This is accomplished by adding electrons supplied by some element with a greater tendency to give up electrons than copper. Zinc is such an element. The net reaction in this case is more clearly written in net ionic form expressing the fact that copper exists in solution as the copper(II) ion and that the negative ions, although present, play no real role in the overall reaction: $Cu^{2+}(aq) + Zn(s) \rightarrow Cu(s) + Zn^{2+}(aq)$. Notice that the electrons required for the reduction of copper ions come from zinc atom. For copper, this reaction represents the opposite of the oxidation that takes place in the first step. This is called **reduction** and we say that the copper(II) ion is **reduced** to copper metal by the zinc. In this same reaction, we can also say that zinc is oxidized to its +2 state. We also refer to zinc as a **more active** metal than copper because it is more easily oxidized.*

There is a secondary reaction taking place as well in this step and it is also related to the fact that zinc is more easily oxidized than copper. Since the formation of copper(II) sulfate took place *in the presence of excess acid, there is a significant amount of H^+ ion present in the solution. Besides being an acid, H^+ ion can also act as an oxidizing agent, accepting electrons from another element, and producing hydrogen gas (H_2). In this secondary reaction, zinc is oxidized by the hydrogen ion: $Zn(s) + 2H^+(aq) \rightarrow Zn^{2+}(aq) + H_2(g)$. Notice that this reaction competes with the first one for the zinc metal and, thereby works against the overall goal of reducing copper to its metallic state. However, with the directions given in this procedure, we should still see most of the copper ion reduced even in the presence of this competing reaction.*

Questions

Answer the following questions before continuing:

What do you observe that indicates that copper(II) ion is being consumed in this step?

What do you observe that indicates that copper metal is being formed?

What do you observe that indicates that the second reaction is taking place (in which the zinc is oxidized by hydrogen ion and making hydrogen gas)?

Step 6: Purification and recovery of copper metal

Decant the liquid from the last step. Add 10 mL of 6 **M** HCl and warm, but do not boil, the solution. The purpose of this step is to oxidize any remaining zinc metal in order to remove it. You may see the production of bubbles of hydrogen gas as this is taking place. When this hydrogen evolution is apparently complete, decant the supernatant liquid, and transfer the copper to a porcelain dish. Use a spatula or rubber policeman. Wash the product with a small amount of distilled water, allow it to settle, and decant the wash water. Repeat the washing and decanting two more times. Move to the hood, away from all flames, and wash and decant with methanol. Place the porcelain dish in the place provided to dry. If this is at the conclusion of a lab period, allow the methanol to evaporate until the following week. If this is at the beginning of a lab period, allow it to evaporate until the end of the period.

Weigh an empty beaker, transfer the purified copper metal into the beaker and weigh again. Compare the final mass to the initial mass of copper used. Determine the percent recovery.

Background information

In step 5, although the oxidation of zinc by the acid competes with the desired reaction, it also helps accomplish another important task. Specifically, if we wish to recover purified copper, we must remove any excess zinc metal from the product. Oxidizing it to soluble zinc ion which can then be rinsed away accomplishes this. In this last step, we add more strong acid in the form of HCl to be certain that this process is complete. All that remains is solid copper and a number of ionic impurities all of which are dissolved in the water which remains. The rinsing and decanting steps help remove these impurities. The purpose of the final rinse with methanol is to help *remove the water. Unless the solution is heated to dry it, the recovered copper metal is likely to be wet. Methanol will remove the water and is much more likely to evaporate completely because it is so much more volatile.*

Questions

Answer the following questions concerning the final step:

> *Assume that, at the end of Step 3, you had only solid copper oxide and water. Considering the reactions which took place in Steps 4, 5, and 6, which ions are present in the water before the rinsing and decanting in Step 6?*
>
> *What percent of the original copper did you recover in the end? If this amount is not equal to 100%, to what do you attribute the difference?*

Summary and conclusions

In this experiment, you saw different forms of the element copper. Summarize the various states of copper that you observed in this reaction by filling out a table such as that shown.

Substance	Formula	Appearance (color)	Water soluble? (Yes/No)
Copper(II) nitrate			
Copper(II) hydroxide			
Copper(II) oxide			
Copper(II) sulfate			
Copper metal			

Your conclusion should summarize in words the results displayed in the table and the reactions that produced these compounds. You should also include balanced chemical reactions for each step. Your observations should be related to the reactions.