Experiment 11 – A Cycle of Copper Reactions

In this experiment, you will perform and observe several reactions of copper. This is a cycle of reactions, because you start and end with the same substance, copper metal. In the first reaction, copper metal is oxidized by nitric acid to form copper (II) nitrate, $Cu(NO_3)_2$. It is then converted to copper (II) hydroxide, $Cu(OH)_2$, by reaction with base. When this compound is heated, it is transformed to copper (II) oxide, CuO. Copper (II) oxide is then reacted with acid to form copper (II) sulfate, $CuSO_4$. Finally, the copper ions in the copper sulfate are reduced to copper metal by magnesium. In theory, you should be able to recover all of the copper that you started with. (The number of atoms of copper is the same throughout all of the reactions.) However, small amounts of copper can be lost during transfers of copper compounds from one container to another. Similarly, small amounts of copper usually get washed away during purification steps. Your goal will be to recover as much copper as possible at the end of the experiment by being very careful in all of the transfers and purification steps that you do.

The unbalanced molecular equations for each of the steps are given below:

1. $Cu_{(s)} + HNO_{3\,(aq)} \rightarrow Cu(NO_3)_{2\,(aq)} + NO_{2\,(g)} + H_2O_{(l)}$

2. $Cu(NO_3)_{2\,(aq)} + NaOH_{(aq)} \rightarrow Cu(OH)_{2\,(s)} + NaNO_{3\,(aq)}$

3. $Cu(OH)_{2\,(s)} \rightarrow CuO_{(s)} + H_2O_{(l)}$

4. $CuO_{(s)} + H_2SO_{4\,(aq)} \rightarrow CuSO_{4\,(aq)} + H_2O_{(l)}$

5. $CuSO_{4\,(aq)} + Mg_{(s)} \rightarrow Cu_{(s)} + MgSO_{4\,(aq)}$

Safety Precautions:

- Wear your safety goggles.
- Concentrated HNO_3 is very corrosive. If it comes into contact with your skin or clothing, wash it off immediately with plenty of water.
- The first reaction must be done under the fume hood. It produces a brown gas, NO_2, which is toxic.
- If any NaOH or H_2SO_4 comes into contact with your skin or clothing, wash it off immediately with plenty of water.
- When using the centrifuge, make sure to place another tube with the same volume of liquid across from your tube. Forgetting to balance the centrifuge can ruin the centrifuge and your experiment.

Waste Disposal:

- All solutions used in this experiment should be disposed of in the **inorganic waste** containers (which have a blue label) in one of the fume hoods.
- Copper metal may be disposed of in the "Waste Copper" container in the hood.

Prelab Questions:

1. Balance each of the equations (if necessary). For each, classify the type of reaction (i.e. gas-forming, decomposition, precipitation, redox, acid-base, etc.). For the redox reactions, state which species is oxidized and which is reduced.
2. Why are you unlikely to get 100% of the metallic copper back?
3. If you were to get more than 100% of the copper back, explain what could have happened (hint: according to the Law of Conservation of Mass, does this make sense?).
4. List the safety rules important for each step.

Procedure

Reaction 1

1. Weigh an empty pointed centrifuge tube on a digital balance, place approximately 0.1 g of copper metal in it, and weigh it again. Record your data.
2. **In the hood**, add 2 mL of 6 M nitric acid. Warm the contents in a hot water bath, if necessary. Heat until the copper has dissolved completely and no more brown fumes (toxic nitrogen dioxide, NO_2) are evolving.
3. Allow the resulting solution to cool to room temperature. (You can put the tube into a beaker of ice water to help it cool off.) Add 2 mL of deionized water to the solution.
4. Record your observations of the reaction, and write the balanced **net ionic equation**.

Reaction 2

5. Add 6 M NaOH drop-by-drop until the solution is basic. Stir well with a stirring rod after the addition of each drop. To tell whether or not it is basic, touch the stirring rod to a piece of red litmus paper. If it turns blue, the solution is basic. (Be careful - don't confuse the blue precipitate with the litmus paper turning blue. You will have to look around the edges of the precipitate at the litmus paper itself.)
6. Record your observations of the reaction, and write the balanced **net ionic equation**.

Reaction 3

7. Gently heat the tube in a beaker of boiling water until a chemical transformation occurs. (The evidence will be a color change.) Continue heating until the reaction is complete. Allow the solution to cool to room temperature on its own (do not use an ice-water bath to cool it).
8. When the solution is cool, centrifuge the tube to collect the solid. (Don't forget to **balance the centrifuge** with a similar tube opposite yours that contains approximately the same volume of liquid.) Decant (pour off) the solution from the tube into a small beaker, leaving the solid behind. (This solution will go into the inorganic waste when you are done.) It is better to leave a little liquid mixed with the solid than to accidentally pour off some solid into the waste. Remember that you want to recover as much copper as possible at the end of the experiment.
9. Wash the solid twice: each time, add 2 mL of deionized water, swirl the centrifuge tube vigorously, centrifuge the tube, and decant the wash water into your waste beaker, leaving the solid behind.
10. Record your observations of the reaction, and write the balanced **net ionic equation**.

Reaction 4

11. Weigh a small beaker that is clean and dry. In this small beaker, place 6 mL of 3 M $H_2SO_{4\,(aq)}$. Using a spatula or a rubber policeman, transfer the centrifuged solid from step 9 into the acid solution. If necessary, rinse the centrifuge tube with a small amount of the sulfuric acid. Rinse the centrifuge tube out with a small amount (1-2 mL) of purified water, and combine all of the rinses in the small beaker.
12. Record your observations of the reaction, and write the balanced **net ionic equation**.

Reaction 5

13. Cut a 30-cm length of magnesium ribbon from the roll of magnesium. Polish the ribbon with fine steel wool until it is shiny. Cut the ribbon into 1-cm lengths.
14. Add about 5 mL of deionized water to the solution in the beaker, and swirl gently. One at a time, add the magnesium strips to the beaker and observe the chemical change that occurs. Keep adding magnesium strips until the solution is colorless. Record your observations of the reaction, and write the balanced **net ionic equation**. If a white, milky precipitate forms ($Mg(OH)_2$), add several drops of 6 M H_2SO_4.
15. Remove a small drop of the solution and add it to some concentrated ammonia in a beaker or test tube. If there are any traces of copper ions still left in the solution, Cu^{2+} and NH_3 will react to form a deep blue complex ion. Observation of this deep blue color indicates that not enough magnesium has been added yet. If you see this color, keep adding magnesium strips to the beaker. (Note: do not remove too many drops of the solution to test for the presence of copper. This step can actually cause you to lose a little bit of copper in each drop you test.)
16. Weigh a clean, dry centrifuge tube and record the mass.
17. With a stirring rod, break up the red-brown coating of copper on the magnesium ribbon. Decant off the liquid into a waste beaker without losing any of the solid. If necessary, transfer all of the solid and some of the liquid into the weighed centrifuge tube, then centrifuge the tube and decant the liquid.
18. Add 6 M H_2SO_4 drop by drop to react with the excess magnesium. In between adding the drops of acid, break up the solid pieces to expose any unreacted magnesium ribbon. Keep doing this until you see no more evidence of reaction. Record your observations of the reaction, and write the balanced **net ionic equation**.
19. If the mixture is not yet in the weighed centrifuge tube, transfer it into the centrifuge tube now. (Transfer all of the solid and some of the liquid.) Centrifuge for 30 seconds, and decant the liquid into a waste beaker. Wash the solid three times with 1-2 mL of deionized water. (Each time: add 1-2 mL water, swirl the tube vigorously to mix it, centrifuge, and decant the rinse water into your waste beaker.)
20. Hold the centrifuge tube with a test tube clamp, and heat it gently over a cool flame, while constantly moving it around. Allow it to cool to room temperature, and then weigh it and record the mass. Again, heat the tube gently for a while, allow it to cool, and weigh it. Keep doing this until the mass is constant. Determine the mass of copper recovered in the experiment.
21. Calculate the percent recovery of the copper.

In your results and discussion section, you should thoroughly discuss reasons for loss of copper at each step of the reaction cycle. Any major errors you make should always be discussed in this section.

Questions:

(Remember - always explain your reasoning.)

1. What is the purpose of adding the strips of magnesium?
2. Why don't we need to weigh the magnesium?
3. For each of the five steps in this procedure, discuss likely reasons for losing or not recovering copper.