Chemical Transformation of Copper

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In this experiment, a weighed amount of copper metal is transformed, through a series of reactions, into other copper-containing compounds, and eventually returned to metallic copper.

The following (unbalanced) equations represent the transformations that will be observed.

A. $Cu(s) + HNO_3(aq) \rightarrow Cu(NO_3)_2(aq) + NO_2(g) + H_2O(l)$
B. $Cu(NO_3)_2(aq) + NaOH(aq) \rightarrow Cu(OH)_2(s) + NaNO_3(aq)$
C. $Cu(OH)_2(s) \rightarrow CuO(s) + H_2O(l)$
D. $CuO(s) + H_2SO_4(aq) \rightarrow CuSO_4(aq) + H_2O(l)$
E. $CuSO_4(aq) + Zn(s) \rightarrow Cu(s) + ZnSO_4(aq)$

Since no copper is added or removed during this series of reactions and since each of these equations go essentially to completion, you should be able to quantitatively recover all the copper you began with. This series of reactions will also show the wide variety of colors often observed for transition metal compounds.

Pre-Lab Assignment

1. Classify each reaction for parts A-E by reaction type (i.e., redox, acid-base, precipitation, or decomposition).

2. Balance each of the reactions for parts A-E.

3. How many milliliters of 6 *M* NaOH are required to react with 0.100 g of Cu to form the $Cu(OH)_2$ in step B? Why is more than this amount added?

4. Based on the balanced equations of question 2, if you start with 0.100 g of copper in part A, calculate the mass of zinc required to complete part E.
   Why is excess zinc added?
   In part E hydrogen gas is also generated. Write the net ionic equation for the generation of hydrogen gas.

Procedure:

Part A: Preparation of Copper(II) Nitrate solution (~45 minutes)

Polish about 1 cm of copper wire with steel wool to remove any oxidation. Wipe the polished wire with a Kimwipe to remove any traces of steel wool. Weigh the wire to the nearest 0.001 g (it should have a mass of 0.080 - 0.100 g) and place it in a 25 mL Erlenmeyer flask. Under the hood, add 2 mL of 6 M HNO_3 and WARM the contents on the stirring hot plate. Do not heat the solution to boiling. NO_2 (a toxic gas) will be generated during the reaction. Record the color of the $NO_2(g)$.
Continue to warm the solution, with frequent swirling, until the copper metal is completely reacted and the evolution of $NO_2(g)$ has ceased. TURN OFF THE HOT PLATE. Remove the flask from the hot plate and allow the solution of copper nitrate to cool to room temperature. Record the color of the solution. Once the solution is cooled add 2 mL of distilled water.

Part B: Preparation of Copper(II) Hydroxide (~10 minutes)

Put a magnetic stir bar into the solution in your flask. Fill a 100 mL beaker approximately half full of tap water and "float" your reaction flask in it. (Your reaction flask should not be sitting on the bottom of the beaker, but no water from the beaker should enter your flask.) Put the beaker and flask assembly on a cool stirring hot plate and adjust the stirring control to achieve a constant motion of the stir bar. DO NOT HEAT. Using a long stem plastic pipet add 6 M NaOH drop wise until the solution is sufficiently basic to form a precipitate of $Cu(OH)_2$. Observe and record the color of the precipitate.

Part C: Preparation of Copper(II) Oxide (~30 minutes)

While continuing to stir with the stir bar, begin heating the beaker/flask assembly until the water in the beaker begins to gently boil. Adjust the heat on the hot plate to maintain this temperature until all the $Cu(OH)_2$ has been converted to black CuO. (If this takes longer than about 3 minutes after the water is boiling, add more NaOH solution.) Remove the flask from the beaker and allow the mixture to cool to room temperature. Remove the stir bar with forceps and rinse the bar with a small (~0.5 mL) amount of distilled water, collecting the rinse in the reaction flask. Isolate the black solid by suction filtration (also called vacuum filtration) using a Hirsch funnel or Büchner funnel which you check out from the stockroom (one per table) and attack to the aspirator in your hood. Remember to put in a piece of filter paper and wet the filter paper with a few drops of water before adding the reaction mixture so that the filter paper sticks to the bottom of the funnel. Rinse the Erlenmeyer flask with 1-2 mL of distilled water from a wash bottle, adding the rinse to the Hirsch or Büchner funnel to insure complete transfer of product. Draw off the filtrate (liquid) by applying suction. Wash the collected solid by adding ~1 mL of distilled water to the solid in the funnel. *If it is difficult to wet the whole sample when washing, then the suction is too strong and you will need to discontinue suction while adding the wash water. Discontinue suction by removing the hose from the flask before turning off the water.* Repeat this final wash procedure with another 1 mL of distilled water.

Part D: Preparation of Copper(II) Sulfate Solution (~10 minutes)

Place 6 mL of 3 M sulfuric acid in a 50 mL beaker. Using a spatula, transfer the black CuO and filter paper to the acid solution. Stir the mixture gently with a glass stirring rod until the black solid has completely dissolved. (Try to keep the filter paper in a few large pieces.) Remove the filter paper from the solution with forceps, and gently rinse it with 1 - 2 mL of distilled water, collecting the rinse in the beaker. If residual traces of CuO remain on the funnel dissolve this material while holding the funnel over the beaker with the solution in the beaker (suck some of the solution up in a plastic buret and squirt it on the CuO in the funnel). Once the transfer is complete, rinse the funnel with 1-2 mL of distilled water, again collecting the rinse in the beaker. The beaker should contain a clear blue solution of $CuSO_4$.

Part E: Regeneration of Copper Metal (~30 minutes)

Working in a hood, slowly add no more than 800 mg of zinc powder or use a piece or two of zinc metal. Stir the mixture with a stir bar until the blue color of the solution disappears (place the beaker over a white background). A vigorous evolution of hydrogen gas is observed and metallic copper will precipitate from the solution during the addition of the zinc. Test the colorless solution for the completeness of reaction by adding 1 drop of the solution to 1 mL of concentrated (15 M) NH_3 solution in a small test tube. If this remains colorless the reaction is complete; if the solution in the test tube turns a deep midnight blue, due to the formation of $[Cu(NH_3)_4]^{2+}$, Cu^{2+} ions are still in solution and the reaction is not complete. If your reaction is not complete continue to stir the solution in your beaker for 2 more minutes and again test for completion. If your reaction is still not complete add additional zinc and stir for another 2 minutes and test for completion.

After the reaction is complete, add 5 mL of 3 M H_2SO_4 solution to the beaker and stir the mixture with a glass rod until the evolution of hydrogen gas ceases. (If you used a piece of zinc metal, remove it and add a few drops of H_2SO_4.) This process removes any unreacted Zn(*s*). Copper metal does not react under these conditions.

Fold a large piece of filter paper into a cone (the instructor will demonstrate) and place in a glass funnel. Pour the copper solid and accompanying liquid through the filter paper. Wash the solid three times with 2 mL portions of distilled water. Carefully remove the filter paper, unfold, and allow the paper and copper to air-dry in your drawer until the next lab period. At the beginning of the next lab period, weigh the copper and calculate the percentage recovery.

Clean the stir bar in 6 M HNO_3.

Post-Lab Questions

1. Which steps would result in a loss of recovered copper and why?
2. Which steps would result in a reported recovered amount of Cu being too large and why?