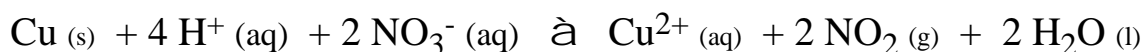
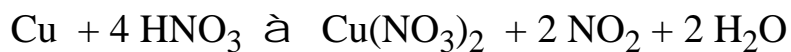


Sequence of Reactions

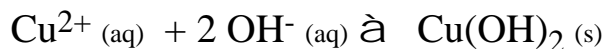
The procedure for the synthesis of a substance may occasionally be relatively complex, involving a sequence of several chemical reactions. In this exercise copper metal, the starting material, is carried through a number of sequential reactions, eventually leading to a final product.

Throughout the process, the student will make observations about the nature and appearance of the products formed. Since matter is neither created nor destroyed in a chemical reaction, the experimenter will be able to make quantitative judgments from the mass of the final product. The reaction sequence utilizes a variety of types of reactions. Chapter 4 of your textbook (Chemistry, 6th Ed, Zumdahl & Zumdahl) provides an excellent description of the major types of chemical reactions. The following introductory material is organized in the order that each reaction is performed, with the type of reaction highlighted.

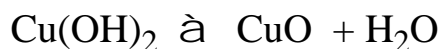
Oxidation Metallic copper may be oxidized by nitric acid. In this reaction, the insoluble metal dissolves as the copper +2 cation. The nitrate anion, NO_3^- is reduced to nitrogen dioxide, NO_2 . The copper (II) nitrate is a strong electrolyte and exists as independent ions in the water solution. The hydrated copper ion in acidic solution has a light blue color.



Precipitation The addition of a strong base such as sodium hydroxide results in the precipitation of insoluble copper hydroxide, $\text{Cu}(\text{OH})_2$. Copper hydroxide is so extremely insoluble that a very fine gelatinous mass is formed which is extremely difficult to filter.

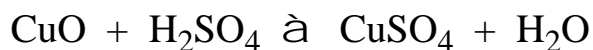


Dehydration The light blue gelatinous copper hydroxide may be converted to black copper oxide simply by heating the solution. The solid copper oxide may then be separated from the solution by filtration.

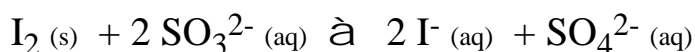
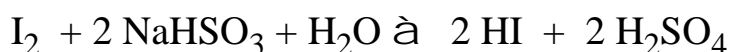
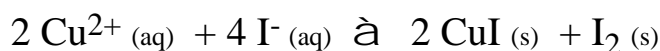
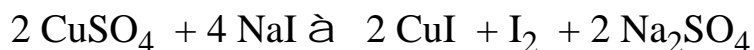


Acid Base Reaction The insoluble, and basic, copper oxide will react with dilute sulfuric acid to

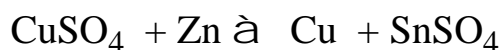
form the very soluble copper sulfate. Copper sulfate is a strong electrolyte and exists as independent ions in solution.



Reduction In the final stage you will be directed by your laboratory instructor to use one of two possible options. In the first option, the copper (II) is reduced by iodide ion to copper (I) and the iodide ion is oxidized to iodine, I_2 . The copper (I) cation then reacts with excess iodide ions to form the insoluble white copper iodide which may then be collected and weighed. The iodine, I_2 , that was formed is also insoluble and must be removed to avoid an incorrect result for the mass of copper iodide. This may be accomplished by adding a second reagent, sodium bisulfite, NaHSO_3 , which will reduce the iodine back to the soluble iodide anion.



The second option involves reducing the copper (II) cations in solution with solid zinc metal to yield metallic copper, which may then be separated from the solution. The unreacted zinc metal must be removed by reaction with hydrochloric acid.



You will be directed to make a quantitative evaluation of this series of reactions from the original mass of copper metal and the final mass of either copper iodide or copper metal.

MATERIALS

from student drawers

- several small beakers
- buchner funnel and vacuum flask
- watch glass
- 100 mL graduated cylinder
- evaporating dish
- reagents from the side bench
 - 10% (w/v) solution of sodium iodide
- zinc metal, 30 mesh
- 6 M hydrochloric acid
- concentrated nitric acid
- dilute sulfuric acid, 15% (v/v)
- 10% sodium bisulfite
 - 20% sodium hydroxide
- pH paper
- filter paper
- common equipment
 - vacuum trap assembly
 - hot plate

SAFETY & DISPOSAL

Concentrated nitric acid is corrosive. Avoid contact by working in a hood and/or wearing gloves. Excess reagents may be disposed of in the laboratory sinks with copious amount of water. Solids should be placed in solid waste containers provided on the side bench.

PROCEDURE

1. Obtain a length of copper wire that weighs between 0.8 and 1.0 grams and weigh to the nearest milligram.
2. Coil the wire into a loose spiral and place it in a 50 mL beaker. Add 10 mL of concentrated nitric acid, cover the beaker with a watch glass and place it in the hood.
3. The copper wire should dissolve completely in five to ten minutes. If it does not, and if the reaction appears to have stopped, add an additional 1 to 2 mL of con HNO_3 , place the beaker on a hot plate and warm.
4. After the solution is complete add 25 mL of water and then slowly add, while stirring, 22 mL

of 20% sodium hydroxide. The mixture should be basic at this point. Check the solution with pH paper to confirm this, and add additional base if necessary.

5. Heat the mixture, stirring to prevent bumping and spattering, until the boiling point is reached. Continue to heat for a few minute to better coagulate the copper oxide and to insure that all of the blue copper hydroxide has been decomposed.

6. Assemble the buchner funnel and vacuum flask assembly as demonstrated by the lab assistant. Filter with suction and wash the precipitate with three 10 mL portions of water. Use the portion of water to rinse the solid copper oxide out of the beaker and onto the filter paper. Discard the filtrate.

7. Transfer the copper oxide to a 150 mL beaker using a small spatula or stirring rod. There may be a small amount of solid which remains on the filter paper.

8. Remove the remainder by pouring back and forth, over the precipitate and through the filter paper, a warm dilute sulfuric acid solution. Add this solution to the beaker containing the copper oxide. Finally rinse the filter paper, funnel and beaker with 10 mL of water and add this to the sample solution.

9. If the copper oxide is not completely dissolved at this point, heat on a hot plate for a few minutes. In some cases a small amount of additional dilute sulfuric acid may be necessary.

10. Option One With stirring, add 50 mL of potassium iodide solution to the blue copper sulfate solution from step 9. Then slowly add, with stirring, the sodium bisulfite solution until all the dark I_2 has been reduced and the precipitate is white. Heat the mixture to boiling for one minute.

11. Suction filter the precipitate through very fine filter paper. It may be necessary to cycle the solution through the paper to insure all the solid has been retained. Dry the filter paper and solid and weigh. Calculate the weight of copper in the copper iodide recovered. Compare this amount with the mass of original copper metal.

10. Option Two While stirring the copper sulfate solution from step 9, add in small portions up to two grams of zinc metal until all of the blue color disappears. Add 10 mL of dilute hydrochloric acid solution to dissolve the excess zinc metal. The solution may be warmed slightly to speed up the reaction.

11. When the zinc is completely dissolved, allow the finely divided copper to settle and decant the solution. Wash the solid with two separate 25 mL portions of water, decanting carefully after each washing.

12. Accurately weigh a clean dry evaporating dish. Transfer all of the copper to the dish, using as little water as possible. Warm the dish until nearly all of the water has evaporated. Remove the dish from the heat when a few milliliters of water remain, and allow the heat in the dish to evaporate the last of the water. Cool and weigh the dish and contents. Calculate the weight of copper recovered and compare this with the mass of original copper metal.

QUESTIONS

1. In step 1 of the procedure, copper metal is oxidized by nitric acid. What is the oxidation number of nitrogen in nitric acid and in nitrogen dioxide.
2. In steps 8 & 9 of the procedure, dilute sulfuric acid reacts with the solid copper oxide. What type of reaction is this?
3. In step 10 (Option One), the reaction of copper sulfate with potassium iodide may be classified as either of two types of reactions. What are these types of reactions?
4. In step 10 (Option Two), the excess zinc metal is dissolved by reaction with dilute hydrochloric acid. What type of reaction is this?