SYNTHESIS AND ANALYSIS OF A COORDINATION COMPOUND OF COPPER

In this experiment you will synthesize a compound by adding NH_3 to a concentrated aqueous solution of copper sulfate. The blue $CuSO_4$ solution will turn a still deeper blue and a mass of small deep blue-to-violet crystals will form as ethyl alcohol is added. On the basis of the analysis of this solid for Cu^{2+}, SO_4^{2-} and NH_3, you will be able to propose a formula for the compound.

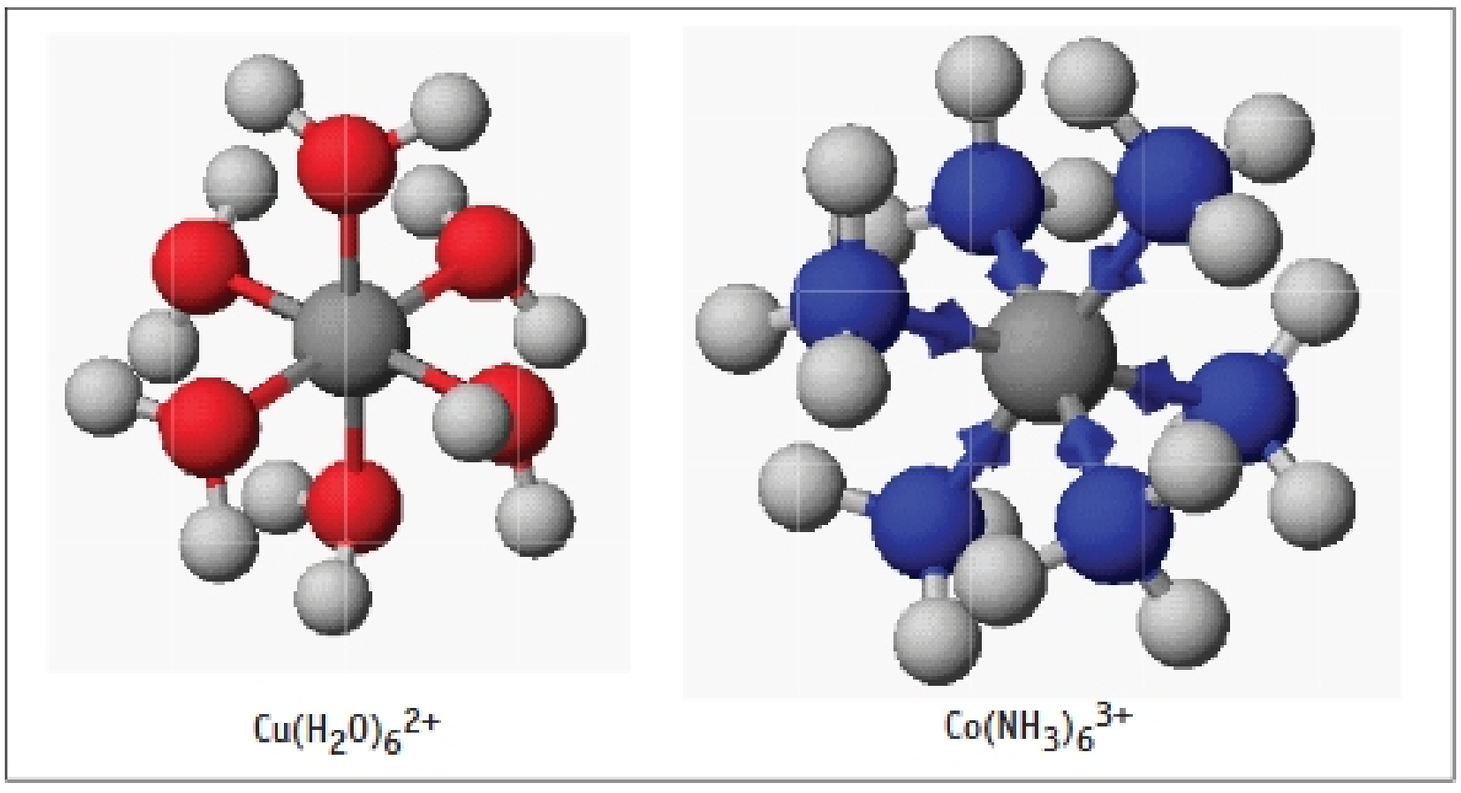
THEORY

Four principal species are present initially in the reaction mixture: copper (II) ions [actually $Cu(H_2O)_6^{2+}$ ions], ammonia molecules (NH_3), sulfate ions (SO_4^{2-}), and water. The product of the synthesis is therefore presumed to be formed by the reaction of two or more of these species. Ethanol is also present, but it is an indirect participant in the reaction. In aqueous solutions ethanol, which is miscible with water but of lower dielectric constant, decreases the solubility of ionic compounds. The marked color change that occurs in the reaction is an important clue to the nature of the product. The product is analyzed for copper (II) ions, sulfate ions, and ammonia molecules. Water is determined as the mass of a sample of the compound that is not accounted for as one of these three species.

The general formula of the unknown compound is $Cu_x(NH_3)_y(SO_4)_z \cdot a\,H_2O$

The analyses to be performed in this experiment are quantitative and are of three types: *gravimetric*, *volumetric*, and *spectrophotometric*. The gravimetric analysis is for sulfate ions; the volumetric analysis is for ammonia molecules; and the spectrophotometric analysis is for copper ions. With careful attention to detail and techniques one can obtain excellent results for each part of the analysis.

You performed gravimetric, volumetric, and spectrophotometric analyses in earlier experiments.



$Cu(H_2O)_6^{2+}$ $Co(NH_3)_6^{3+}$

Complex ions containing bound water and ammonia. The structure of Cu^{2+} ion in water is on the left. Note that the water molecules are bound, through oxygen atoms, to the copper ion. In this experiment you will make a compound that has NH_3 molecules bound to the copper(II) ion through N atoms, similar to the complex ion $[Co(NH_3)_6]^{3+}$.

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PROCEDURE

A. Synthesis of the Compound

1. Weigh out 10.0 grams of copper sulfate pentahydrate, $CuSO_4 \cdot 5H_2O$, on a triple beam balance, and place the crystals in a 250 mL beaker.
2. Add 10-15 mL of water to the solid and then in a hood add 20 mL of 15 M NH_3 (concentrated ammonia). Stir to dissolve the crystals as ascertained by holding the beaker up to the light.
3. Over a period of 1 minute, slowly add 20 mL of 95% ethanol (ethyl alcohol) to the solution, stir, and cool to room temperature.
4. Meanwhile, prepare 30 mL of a solution from 15 mL each of concentrated ammonia and 95% ethanol. Cover all your solutions containing ammonia with watch glasses (to prevent the fumes of NH_3 from saturating the lab atmosphere), and return to your desk.
5. Set up an apparatus for *vacuum filtration*. Moisten the filter paper and turn on the aspirator. Carefully filter the slurry of crystals that has formed in the copper-containing solution and suck off all the solution.

> In the event that a significant amount of crystalline product remains in the beaker, you should use the filtrate from the filter flask to wash the crystals onto the filter paper. To prevent backup of tap water into the filter flask, which would render the filtrate useless as a wash, pull the hose off the aspirator while the water is still running. Remove the filter funnel and pour the filtrate from the filter flask back into the beaker that contains the crystals. Reassemble the filter apparatus and collect the remaining product on the filter paper.

Turn off the aspirator and carefully pour 10 mL of the ammonia-ethanol solution onto the crystals. Break up all lumps of solid to permit the liquid to penetrate the mass completely and then turn on the aspirator to suck off the liquid. Repeat the washing procedure twice with additional 10 mL portions of ammonia-ethanol. Next wash twice with 10 mL portions of 95% ethanol and finally with two 10 mL portions of acetone, breaking up the mass of crystals with a spatula in each step before turning on the aspirator. (At this point your crystals should appear nearly "dry," that is, not moistened with liquid to any great extent. If you feel they could be dried more, add additional amounts of acetone as described above.) To remove the last traces of moisture and other solvents from your solid, draw air through the crystals for at least 5 minutes, using your spatula to break up any remaining lumps.

CAUTION: acetone is flammable

6. Put the crystals in a large test tube or small beaker, lightly cover, and leave them in your desk to dry thoroughly until the next laboratory period.
7. When you come to the laboratory period following the one in which the crystals were prepared, inspect them to insure they are dry and then weigh the entire sample to the nearest 0.1 gram. Record the mass on the report form, and then go on to the next portion of the experiment.

ANALYSIS OF THE COMPOUND

A. Gravimetric Analysis for Sulfate Ion, SO_4^{2-}

1. Weigh to the nearest milligram about 0.9-1.1 gram of your compound (after making absolutely sure it is dry).
2. Dissolve the solid compound in 10 mL of 6 M nitric acid (HNO_3). The reaction occurring at this point is

   $$Cu_x(NH_3)_y(SO_4)_z \cdot a\ H_2O + y\ H^+ \rightarrow x\ Cu^{2+} + y\ NH_4^+ + z\ SO_4^{2-} + a\ H_2O$$

3. Obtain 5-6 mL of 1 M lead acetate [$Pb(C_2H_3O_2)_2$], and add it dropwise to the acid solution until precipitation is complete. The reaction now occurring is

   $$Pb^{2+}(aq) + SO_4^{2-}(aq) \rightarrow PbSO_4(s)$$

During addition of nitric acid to the unknown compound in water the originally deep violet solution will turn light blue.

Use the following procedure to check for completeness of precipitation: First allow all the white precipitate to settle to the bottom of the beaker. Using your eyedropper, allow one drop of lead acetate solution to run down the side of the beaker into the light blue solution. If precipitation of the SO_4^{2-} ion is not yet complete, you will see a white precipitate of $PbSO_4$ form just as the drop slides down the beaker wall and into the solution. If you do not observe a white solid forming at this point, then precipitation is complete and you can proceed.

4. Once you have decided that precipitation of the $PbSO_4$ is complete, weigh a piece of filter paper to the nearest milligram. Assemble the apparatus for *gravity filtration*. After properly folding the paper into a cone, place it in the funnel, moisten it with a little water, and adjust it so that it fits the funnel snugly. Be careful not to tear the paper. Using a stirring rod to guide the flow of liquid, carefully fill the filter cone about one-half full of the lead sulfate-water slurry. When this has drained nearly empty, repeat the operation, and continue this process until the transfer of lead sulfate to the filter is complete. It is important that you do not lose any of the precipitate in the transfer to the filter paper.

Recall that you did a gravity filtration when making an iron oxalate complex in the first experiment, when making alum, and then doing the gravimetric analysis of a barium salt.

5. The filtration described above may be quite slow. Since you must do a duplicate determination, you may wish to start the second one while the first one is filtering.
6. After most of the liquid has collected from the filtration, check it once again for completeness of precipitation by adding a few drops of lead acetate. If a white precipitate forms (the solution becomes cloudy), add another milliliter of lead acetate solution and refilter.
7. Rinse the beaker with small portions of water and use these rinsings to wash the precipitate on the filter paper. Finally use your wash bottle to rinse the filter paper and precipitate free from the original copper-containing solution.
8. Rinse the precipitate with a small portion (say 10 mL) of acetone.
9. When the liquid from the last washing has drained out, remove the filter paper (be careful not to tear it!) and place it in a 250 mL beaker or evaporating dish to dry until the next laboratory period. When you are certain

Acetone, H_3COCH_3, forms hydrogen bonds with water. This assists in removing water from the precipitate and thereby hastening its drying.