Determining the Percent Composition and Formula of a Copper Chloride Hydrate

Overview:

The mass percents of Cu, Cl and H₂O in a compound are determined by separating and massing the three components.

A major emphasis of laboratory work for a chemist is determining the composition of a compound. There are many tools (such as, chromatographic separation and spectroscopy) available to aid the chemist in determining chemical composition. As you study chemistry at Wofford you will be exposed to many of these analysis techniques. In this lab you will become familiar with the techniques of sample heating, vacuum filtration, and the proper handling of strong acids.

We will start with a copper chloride hydrate of unknown composition (CuₓClᵧ • z H₂O). We will sequentially separate the components of water and copper, leaving behind a solution of chloride ions. The water will be removed by heating. Hydrated salts quickly lose their waters of hydration during heating. Reweighing the sample after heating will reveal the mass of anhydrous (without water) CuₓClᵧ, and thus the mass of water initially in the compound. Dissolving the salt in water and reacting the copper ions with a more reactive metal will separate out the copper. The copper collected in this way is then massed. The mass of chloride present in the compound is obtained by difference.

Experimental Procedure:

Removing the H₂O:

- Turn on the hotplate located between the desks or in the hood and adjust to a setting of 5 or 6.
- Clean, dry, and weigh a small ceramic crucible.
- Weigh out between 1 and 2 grams of the blue copper chloride hydrate into the crucible. Remember, no chemical is to be dispensed at the balance. Obtain the mass of the crucible and compound together.
- Heat the sample in the crucible to drive off the water. Watch the heating carefully. If the sample begins to spit as it releases water, remove the sample from the hot plate with your tongs. If the hot plate is too hot, lower the temperature.
- As the water is removed from the compound, the color will change from blue to brown. Continue heating until the blue color is gone. You may need to gently stir the powder with your spatula. Any compound that sticks to the spatula should be taped back into the crucible. Heat until no more color change is observed.
- Remove the crucible from the hot plate with crucible tongs. Let the sample cool with the lid in place. On humid days the salt will start to rehydrate from water in the air. You cannot weigh a hot object on the balance. The heat will cause convection currents over the balance, resulting in unstable readings.
- After cooling (about 5 minutes), weigh the dehydrated sample and crucible.
• Have you removed all of the water from the sample? It is difficult to tell. Reheat the sample for five minutes. Let the sample cool on the desk and weigh. If the mass has changed more than 0.005 grams, you are still driving off water and should continue to heat in five-minute intervals.

• If you are the last to use the hotplate at your station, turn it off.

Collecting the Copper:

The copper in the sample (Cu₂Cl₂ at this point) will be removed by an oxidation/reduction reaction with aluminum. The copper ions will be reduced by the aluminum to form copper metal. In the language of oxidation/reduction we would say that the aluminum metal is being oxidized to aluminum ions. We will investigate the details of oxidation and reduction in Chapter 4.

• Transfer the copper chloride salt to a 100 ml beaker and dissolve the salt in 50 mL of water. Use your squirt bottle to rinse the crucible, making sure that all of the compound transfers from the crucible to the beaker.

• Once the compound is dissolved, the blue copper ions are floating freely in the solution. Obtain approximately 0.15 g of aluminum mesh. Add the aluminum to the copper solution. You must stir vigorously during the reaction. As the copper ions are reduced, their blue color will fade from the solution and a brown copper solid will appear in the solution. If all of the aluminum has been oxidized (reacted) and a blue green color remains, add a few more strands of aluminum.

• In the end, we must have more aluminum present than there is copper, to ensure that all of the copper has been reacted. However, we also want the only solid present to be copper. After the blue copper color is removed (approximately 30 minutes of reaction), the excess aluminum solid can be removed by continuing the oxidation process with HCl.

• If there are large chunks of aluminum in the solution, these can be removed with forceps. As you extract aluminum use your squirt bottle to remove any copper flakes clinging to the aluminum. You want to collect all of the copper.

• Remove the excess aluminum in solution by adding several mL of 6M HCl to the solution. Hydrogen gas is released during the reaction between aluminum and HCl. Stir the solution and continue adding small amounts of HCl until the hydrogen bubbling ceases.

• After the aluminum has been dissolved in solution you will separate the copper by vacuum filtration in a Buchner funnel. The vacuum filtration is set-up in each hood. Obtain a piece of filter paper. Record the mass of the filter paper on a watch glass. After collecting the copper, you will weigh the copper, filter paper and watch glass to determine the mass of copper.

• Place the filter paper in the Buchner funnel. Attach the vacuum hose to the vacuum connection in the hood. Squirt down your filter paper with DI water from your squirt bottle. Turn the vacuum on at the side of the hood. Turn the vacuum on full blast. There is no reason to just have a little vacuum. The water in the funnel should be pushed straight through into the filter flask and the filter paper should make a good seal with the bottom of the funnel. If the water does not quickly drain when the vacuum is applied you have a leak. Disassemble the apparatus and reconnect the pieces to eliminate the leak.
• In filtering, your goal is to collect all of the brown copper onto the filter paper. Gently swirl the solution so that the copper is no longer on the bottom of the beaker. Pour a large quantity of the solution into the funnel (not so much that you overfill the funnel). After the funnel drains, add the remaining solution. Use your squirt bottle to extract any additional copper stuck to the sides of the beaker.
• Once all of the copper you can see is on your filter paper, you need to remove as much of the water on the paper as possible. Most of the water can be washed away with ethanol that is much more volatile (and will evaporate quickly). Obtain 10 mL of 95% ethanol from the front of the room in your smallest graduated cylinder. Pour this over the copper and filter paper. Make sure everything in the funnel is rinsed with the ethanol. Let the vacuum run for three minutes after the ethanol wash.
• Turn the vacuum off and remove the plastic top of the Buchner funnel. (If you have a ceramic funnel remove the whole funnel.) At your lab desk use your spatula to gently extract the filter paper, placing it on the watch glass. Remember that you want to get as much copper as you can onto the watch glass. If any aluminum wire is present, pick it out with forceps. Empty the solution in the bottom of the filter flask into the aluminum waste container. Reassemble the funnel and filter flask for the next group.
• Place the watch glass with filter paper and copper in the oven. Notice where in the oven you have placed your sample. The ethanol will be vaporized from the sample in about five minutes.
• Using a heat glove, remove your sample and let it cool on your desktop.
• Weigh the cool, dried sample to obtain the mass of copper recovered.
• Once the mass of copper has been determined, the filter paper and copper can be placed in the regular trash can.

**Determination of the chloride:**

• The mass of chloride in the sample is obtained by difference.

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\text{Cl mass} = \text{sample mass} - \text{H}_2\text{O mass} - \text{Cu mass}
\]

**Determining Chemical Formula:**

From your mass measurements determine the mass percent of Cu, Cl and H\(_2\)O in the sample.

Reporting the mass percent of each species in a compound is extremely useful, but we would also like to know the ratio of the atoms/moles in the sample. The problem converting mass percent to atom percent is that copper weighs more than water (it just has more protons and neutrons). You, in fact, need to have 3.5 waters to have the same mass as one atom of copper. The atom percent in a sample is what we know as the chemical formula and it is given as the simplest whole number ratio for ionic compounds (ex. CrCl\(_3\) • 6 H\(_2\)O).

To obtain the atom/mole ratios (chemical formula) from the mass percent, you need to (1) assume 100 g of sample so that percentages give gram quantities of each component; then (2) divide each mass by the molar mass of each component. For example a nickel (Ni) phosphate (PO\(_4^{3-}\)) compound is found to be 48.1 % Ni and 51.9 % PO\(_4^{3-}\) by mass. This compound is not
half nickel and half phosphate as the mass percents indicate. Rather, in a 100 gram sample there is 48.1 g Ni and 51.9 g PO$_4^{3-}$. Dividing the mass of each component by their molar masses (Ni = 58.7 g/mol, PO$_4^{3-}$ = 95.0 g/mol) gives their molar (atomic) composition.

$$48.1 \text{ g Ni} \left( \frac{\text{mol Ni}}{58.7 \text{ g Ni}} \right) = 0.819 \text{ mol Ni} \quad \text{and} \quad 51.9 \text{ g PO}_4^{3-} \left( \frac{\text{mol PO}_4^{3-}}{94.9 \text{ g PO}_4^{3-}} \right) = 0.547 \text{ mol PO}_4^{3-}$$

It is interesting that there is actually more nickel in the sample than phosphate. The atomic composition of this compound is then Ni$_{0.819}$(PO$_4^{3-}$)$_{0.547}$. (3) This non-integer representation of the atom ratios is exceedingly ugly and should be converted to whole numbers by dividing each amount by the smallest amount present.

$$\frac{\text{Ni}}{\text{PO}_4^{3-}} = \frac{0.819}{0.547} \approx 1.5$$

We still find that atom quantities are not expressed as integer values. (4) We must find a common multiplier that will result in integer values of each atom. In this case the multiplier is two. Multiplying each atom amount by two yields the empirical formula

Ni$_3$(PO$_4$)$_2$

The above formula for nickel (II) phosphate would be universally recognized by all scientists.

(1) Convert your mass percents for Cu, Cl and H$_2$O into grams for a 100 g sample; then into mole ratios by (2) dividing the percentage by the molar mass of the respective atom or molecule. If necessary, convert these numbers into the smallest whole number ratios by (3) dividing each amount by the smallest amount present and (4) finding a common multiplier that will result in integer values of each atom or molecule. Report the whole number ratios in your notebook.