

Chemistry 112 Laboratory – Fall 2004

Experiment 3: Determination of a Chemical Formula

Overview

In this experiment you will be analyzing a compound with the general formula $\text{Cu}_x\text{Cl}_y \cdot z\text{H}_2\text{O}$, where x , y , and z are integers which, when known, establish the formula of the compound. This compound is an example of a *hydrated crystal*, in which (intact) water molecules are incorporated into the crystal structure of a solid salt. How many water molecules fit into each formula unit (represented by the variable z) depends on the geometry of the crystal; but it is almost always a whole number. Hydrated crystals often differ markedly in appearance from their anhydrous ($z=0$) equivalents, and that will be seen to be the case in this experiment. In expressing the formula of a hydrated crystal like this one, because the water molecules remain intact, we indicate the number of water molecules incorporated into the crystal (per formula unit) with the nomenclature $\cdot z\text{H}_2\text{O}$, rather than adding H_2O to the formula of the salt itself.

The compound we will study, a copper chloride hydrate, turns out to be really nifty for one's first venture into formula determination. It is stable, can be obtained in pure form, and has a characteristic color which changes as the compound is dehydrated. It is also relatively easy to decompose into its component elements and water. In this experiment we will first drive off the water (the "water of hydration") from an accurately weighed sample of the hydrated salt. This occurs if we gently heat the sample to a little over 100°C . As the water is driven off, the color and consistency of the sample changes. The resulting compound is anhydrous (no water) copper chloride. We then subtract its mass from that of the hydrate we started with, which allows us to determine the mass of the water that was driven off, and, using the molar mass of water, find the number of moles of H_2O that were in the sample.

In the next step we need to find either the mass of copper or the mass of chlorine in the anhydrous sample we have prepared. It turns out to be much easier to determine the mass of the copper, and to then find the mass of chlorine by difference. We do this by dissolving the anhydrous sample in water, which gives us a green solution containing copper and chloride ions.

To that solution we add some alumin(i)um metal wire. Alumin(i)um is what we call an active metal; in contact with a solution containing copper ions, the alumin(i)um metal reacts chemically with those ions, converting them to copper metal. The alumin(i)um is said to reduce the copper ions to the metal, and is itself oxidized. The copper metal appears on the wire as the reaction proceeds, and has the typical red-orange color of copper. When the reaction is complete, we remove the excess Al, separate the copper from the solution, and weigh the dried metal. From its mass we can calculate the number of moles of copper in the sample. We find the mass of chlorine by subtracting the mass of copper from that of the anhydrous copper chloride, and from that value determine the number of moles of chlorine. The mole ratio for Cu:Cl:H₂O gives us the *empirical* formula of the compound, which incorporates the simplest whole number ratio of the values x, y, and z. (The actual values may be 2x, 2y, and 2z, or 3x, 3y, and 3z, or some other whole-number multiple of the empirical formula.)

Note Bene: In the interest of world harmony, the correct and incorrect spelling of alumin(i)um are both referenced in this document. Far be it from me, as the son of an Italian and German, a former Canadian citizen born and trained in the US, but having used the word for this handy metal most extensively in the former British colony of Fiji, to say which spelling is the correct one. Can't we all just get along? =)

Procedure

Weigh a clean, dry crucible, without a cover, accurately on the analytical balance. Place 1.0 g of the unknown hydrated copper chloride into the crucible. With your spatula, break up any sizeable crystal agglomerates by pressing them against the wall of the crucible. Then weigh the crucible and its contents accurately on the analytical balance, recording the mass to ± 0.0001 g.

Place the uncovered crucible on a clay triangle supported by an iron ring. Adjust the ring so that it sits about 10 centimeters above the top of the Bunsen burner. Light your Bunsen burner away from the crucible, and adjust the burner so that you have a small flame, four or five centimeters high, but blue rather than yellow. Holding the burner in your hand, gently heat the crucible as you move the burner back and forth about 10 centimeters underneath it. *You don't want to overheat your sample, or it will turn into a fused-solid black mass and you will have to start over!* As the sample warms, you will see that the crystals begin to change around the edges. Continue gentle heating, slowly converting all of the hydrated crystals to the anhydrous form. After all of the crystals appear to have changed, continue heating gently, moving the burner back and forth judiciously, for an additional two minutes. Remove the burner, and cover the crucible to minimize rehydration. (The anhydrous salt will draw water out of the air and return to the hydrated form, if you let it!) Let the crucible and its contents cool for about 15 minutes. [While you are waiting, write down what you just did, and what you saw! If you have time, you might want to check that the test tubes and other glassware you used last week are clean.] Remove the crucible cover, and slowly roll the brown crystals around in the crucible. If any green (hydrated) crystals remain, repeat the heating process. (If any significant black globs are stuck to the bottom of your crucible, consult with your instructor.) Finally, determine the mass of the cool, uncovered crucible, and its contents, accurately, with an analytical balance.

Do not proceed until you have determined the mass of the anhydrous salt, or you'll have done the steps above for nothing! Once you have the data you need to determine the mass of the anhydrous salt, you can transfer the anhydrous salt in the crucible to an empty 30 or 50 mL beaker. Rinse out the crucible with two 6 mL portions of distilled water, and add the rinsings to the beaker. A very little bit of the copper salt may remain stuck to the crucible, but the

amount should be minimal. Consult with your instructor if it is not! Swirl the beaker gently to dissolve the solid. The color will change as the copper ions are re-hydrated. Measure out 20. cm of 20-gauge alumin(i)um wire and form the wire into a tight coil by wrapping it around one of your large test tubes. Pull about two centimeters of the wire up and out of the coil, so that when you put the coil into the solution, it is completely immersed, with the exception of this little "handle." Within a few moments you will observe some gas evolution, as well as the formation of copper metal on the Al wire. As the copper ions are reduced, the color of the solution will fade. The Al wire will be slowly oxidized and enter the solution as alumin(i)um (Al^{3+}) ions.

When the reaction is complete, which will take about 30 minutes, the bubbling will stop, the solution will be colorless, and much of the copper metal that was produced will be on the Al wire. You can speed up the reaction somewhat by periodically (every 5 to 10 minutes) jiggling the Al wire by its handle, vigorously enough that some of the copper comes off of it. (*While you are waiting for the reaction to finish up, you should write down what you have done and what you have seen. You will probably also have time to work out the amount of water that was present in the hydrated salt!*) Once the reaction is complete and the solution no longer has a green tint, use plastic tweezers and your stirring rod to remove the precipitated copper from the wire as completely as you can. Pull the alumin(i)um wire out of the beaker and, while the wire is suspended above the beaker, rinse off any remaining Cu particles with forceful squirts of water from your wash bottle. (Go ahead and squirt hard – if your squirt bottle isn't up to the task, get a different one!) Complete the removal of Cu with five drops of 6 M HCl added directly to the wire while it is held so that the coils are horizontal. It'll fizz! Let it do so for a little while, trying to keep the HCl from dripping off the wire immediately. Follow this with a final rinse from the squirt bottle. Put the (now clean) alumin(i)um wire aside; it has done its duty! You can recycle it in the container designated for this purpose.

In the beaker you now have the metallic copper produced in the reaction, in a solution containing an alumin(i)um salt. Set up a small Büchner funnel over a 250 mL sidearm flask, clamping the flask in place and connecting it to the vacuum line with vacuum hose. Fit the funnel with a circle of Grade 6 filter paper. Squirt the filter paper with distilled water to moisten it

completely, and then turn on the vacuum line just enough that you hear air flowing. Before the filter paper dries, decant the solution into the funnel. (Don't worry if some of the copper is also transferred.) Wash the copper metal in the beaker with small portions of distilled water; decanting the wash into the funnel. Break up any copper particles with your stirring rod, and wash again, twice. Transfer the wash and the copper to the filter funnel. Wash any remaining copper in the funnel with water from your wash bottle. *All* of the copper must be transferred to the funnel. Rinse the copper on the paper once again with water. Turn off the suction. Add 10 mL of 95% ethanol to the funnel, and after a minute or so turn on the suction. Draw air through the funnel for about 5 minutes. Transfer the copper from the funnel to a watch glass whose mass has been accurately determined; this is perhaps most easily done by lifting the edge of the filter paper with a spatula and then lifting the paper from the funnel. The transfer must be quantitative; scrape any copper that adheres to the paper onto the watch glass with your spatula. Dry the copper on the watch glass under a heat lamp for 5 minutes. Allow it to cool to room temperature and then accurately determine its mass.

Analysis

You should calculate the empirical formula of the unknown salt, using the data you collected in the lab. Compare your result with that of your classmates, to make sure you aren't making any silly mistakes! You may also want to look up the possible forms that hydrated copper chlorides might take, in a reference book, like the CRC Handbook of Chemistry and Physics, Lange's Handbook of Chemistry, or the Merck Index. Keep in mind that you got a glance at the *anhydrous* salt with the formula Cu_xCl_y , so you might want to look at possibilities there, too, and determine if what you saw makes an optimal match to one of them.

Once you have a best guess at the identity of your unknown salt, you can calculate a theoretical formula weight for it, and from that the theoretical mass of water and copper you should have obtained in your analysis. Compare these with your actual results, calculating your percent yield for each case. In each case, provide one or two (not more!) *plausible*, ideally even likely, explanations for why your yield in was not exactly 100%. Think through the consequences

of your proposed source of error, to be sure that it would tilt your results in the direction they actually tilted! For example, it would be patently incorrect to argue that you got 104% yield in your water extraction because you failed to completely dehydrate your unknown! Failing to drive off all the water of hydration would result in a yield of *less than 100%* for that part.

Reporting

This lab is the first of three for which you will be writing a formal report. (Yummy!) You'll get details on that separately, from Professor Kuwata. You should still keep excellent notes and carry out all your calculations in your lab notebook, though, both because it will be graded separately and because it will provide the grist for your efforts to assemble the formal report!

Advance Study Assignment

Experiment 3: Determination of a Chemical Formula

1. In the lab book you will use for this experiment, (Lab Book I), add this experiment to your table of contents, and write the title on the page where the experiment will start.
2. Next, write a short introduction that provides an overview of what you understand as your objective for this experiment, and what you understand the fundamental steps to be. It should be a few sentences long.
3. After your introduction, under the heading "Advanced Study Assignment Calculations," do your best to solve the following problem. Show your work in your lab notebook, and do it *all* there. Do not attempt to make your mistakes vanish, in any way. Yes, really! Be mindful of significant figures, and remember that

$$\text{yield} = \frac{\text{theoretical yield}}{\text{actual yield}} \quad \text{error} = \left| \frac{\text{theoretical value} - \text{actual value}}{\text{theoretical value}} \right|$$

A molecule being considered as a drug candidate ($C_xH_yO_z$) consists of only carbon, hydrogen, and oxygen. The molar mass of this substance was determined to be 33_4 g/mol in a freezing point depression experiment (à la Example 8.8 on page 311 in your book). [Freezing point analysis of a pure substance provides an accurate, but not exceptionally precise, molar mass. It's not more than 5% off.] A combustion analysis was attempted to elucidate the molecular formula of this substance. Using the apparatus described in Figure M.1 on page F91 of your textbook, 2.253 grams of the drug candidate was ignited in an oxygen-helium mixture and 6.03_5 g of carbon dioxide and 1.48_1 g of water vapor were detected with the gas chromatograph/thermal conductivity system.

- a. Determine the *empirical* formula of the drug candidate.
- b. Determine the *molecular* formula of the drug candidate.
- c. Based on the "correct" molar mass of your answer in (b), calculate
 - i. The % error in the molar mass value given in the problem
 - ii. The % yield of carbon dioxide in the combustion analysis