Determining the Percent Composition of a Copper Chloride Hydrate

Overview:

The mass percents of Cu, Cl and H_2O in a compound are determined by separating and massing the three components. The resulting data is then used to determine the empirical formula of the hydrated salt.

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, absorbance spectroscopy and the proper handling of strong acids.

We will start with a copper chloride hydrate of unknown composition ($Cu_XCl_Y·ZH_2O$). We will sequentially separate the components of water, copper ions and 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) Cu_XCl_Y and thus the mass of water initially in the compound. Dissolving the salt in water will produce copper ions and chloride ions. The copper ions in water are an aqua blue color. The amount of copper present in the sample will be determined by a spectroscopic technique called colorimetry. The mass percent of chloride present in the compound is obtained by difference.

Experimental Procedure.

Work in lab groups of two for the first part of the lab. When it is time to use the spectrometer at your lab table, the lab groups will work together.

Removing the H_2O:

- Turn on the hotplate located between the desks and adjust to a setting of 8 or 9.
- Clean, dry and weigh a small ceramic crucible from your lab drawer.
- Weigh out between 1 and 1.5 grams of the blue copper chloride hydrate into the crucible. Record the mass of the sample to four digits. **Remember, no chemical is to be dispensed over the balance. Obtain the mass of the crucible and then weigh the crucible and compound together.**
- On the hot plate, 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 and lower the hot plate temperature. *Why do you imagine that this might be of importance?*
- As the water is removed from the compound, the color will change from blue to brown. Continue heating until the blue color is gone. With your tongs, tap or rattle the crucible to expose the copper chloride hydrate in the middle. Heat until no blue/green crystals are observed. This will take ten to fifteen minutes.

- During heating, a member of the lab team should begin work on the solution preparation for the copper determination (see next page) and wavelength vs color of light.
- Once all crystals are brown, 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 for five minutes, mass the dehydrated sample.

Have you removed all of the water from the sample? It is difficult to tell. To check for water removal, 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. (Your instructor may have you skip this second heating step if there are no blue crystals in your sample.)

- If you are the last to use the hotplate at your station, turn it off.
- From the mass decrease determine the grams of H_2O in the original $Cu_XCl_Y \bullet ZH_2O$ sample.
- Determine the % mass of H_2O in this compound.
- Transfer the brown Cu_xCl_y sample from your crucible to the solid waste jar in the hood. Rinse and dry the crucible.

Quantifying the Copper:

The amount of light absorbed by a sample is dependant upon the nature of the sample itself, the wavelength of light used in the experiment, and the concentration of the solution being tested. Your task here will be three fold:

- Determine the color of light associated with the wavelengths of light our colorimeters can measure.
- Determine the wavelength of light that is best absorbed by aqueous solutions of copper ions.
- Prepare solutions of known concentration of copper ions and measure their absorbances.
- Prepare a calibration plot of absorbance vs concentration of copper ions
- Prepare an aqueous solution of your unknown, measure its absorbance, and read the concentration of copper ions from your calibration curve.

Wavelength of light vs color:

- Prepare a table in your lab notebook for recording your results
- Open the cover to your colorimeter. Place a strip of cardstock in the colorimeter so that it leans towards the front of the instrument.
- Select each of the four wavelengths, one at a time, and record the color that you see on the bottom of the cardstock.

Preparation of Copper Chloride Hydrate Solution / Determining Which Wavelength to use.

The amount of copper present in the compound $Cu_XCl_Y \cdot ZH_2O$ will be measured by examining the amount of light absorbed by the sample in solution. This, in some respects, is similar to the solution density vs. sugar concentration calibration plot you made last week. You will prepare an absorbance vs. copper concentration plot using prepared solutions of known copper concentration.

- Obtain a 25 mL volumetric flask from your teaching assistant. Transfer several micro spatula scoops of the copper chloride hydrate salt to the 25 mL volumetric flask. You need to have between 0.3 and 0.5 grams of the **hydrated** salt added to your volumetric flask. It is important that you know the exact weight of the $Cu_XCl_Y \cdot ZH_2O$ added, so make mass measurements of the flask before and after the addition of the $Cu_XCl_Y \cdot ZH_2O$.
- Dissolve the solid by filling the flask half full and swirling. After dissolving the solid, fill the volumetric flask containing the know mass of $Cu_XCl_Y \cdot ZH_2O$ to the 25 mL line with DI water using your squirt bottle. Use a dropper to add the last few drops of water. If you go past the 25 mL mark, you will not know the volume of your sample and you will need to make a second copper solution. Cap the flask and invert the solution (30 – 40 times) so that the solution is of uniform concentration.

When the compound has dissolved, the copper ion is complexed to four water molecules to form a solution. What color is that solution? The chloride ions are colorless. The color of the solution is due to the copper ions. The ions absob light because an electron moves from water to the copper ion. We will study electronic structure later in the semester. We shall take advantage of this property of copper ions in water to determine the amount of copper in your solution. The more copper ions in solution, the deeper the color. We will use a type of spectrophotometer (a colorimeter) to measure how much light is absorbed by the copper ions. Then we will compare this to the absorption characteristics of copper solutions of known copper concentration.

- Half fill the plastic cuvette with your aqueous copper hydrate solution, pour this solution out into the waste beaker at your desk and repeat two more times.
- Fill the cuvette about 3/4 full with your aqueous copper choride solution, wipe the outside of the cuvette clean, and insert it into the sample holder of your colorimeter. (Do remember to remove the card stock first.)
- Record the absorbance at each wavelength of light that your colorimeter will measure.
- Tabulate your results.

Which wavelength of light gave the highest absorbance reading on the colorimeter? What color of light does that wavelength correspond to? The wavelength of light that gave the highest absorbance reading will be the best one to use in today's experiment as that will give you the most sensitive test for copper ions in solution.

<u>Preparation of Standard Solutions of Copper Ions</u>

Begin working with everyone at your lab table to prepare the copper standard solutions. Be sure that everyone prepares at least one solution using the volumetric glassware. There will be several copper solutions on your table, so it is important to develop a labeling scheme to keep these solutions properly identified.

- Your lab group has prepared its own copper solution of unknown composition and now collectively your lab table (which is made up of two lab groups) needs to prepare copper solutions of known concentration. At the front of the room is the copper salt $CuSO_4 \cdot 5\,H_2O$ [copper(II) sulfate pentahydrate]. Measure about 0.4 grams of $CuSO_4$ exactly and place it into one of your table's 25 mL volumetric flasks. To do this you should first mass the empty flask and then away from the balance add $CuSO_4 \cdot 5\,H_2O$ a little bit at a time. Wipe the spatula on a paper towel before returning it to the reagent bottle. Wipe the top of the volumetric flask with a paper towel before taking your final mass measurement. Be sure to record your masses to the thousandth decimal place. Dispose of your dirty paper towel in the trash.
- Fill the volumetric flask to the 25 mL mark with DI water. Use an eye dropper to add the last few drops of water. You now have a copper standard that is close to a copper concentration of 0.1 g Cu / (25 mL H_2O). From the exact mass of $CuSO_4$ $\cdot 5H_2O$in your sample, calculate the grams of copper in your sample to three decimal places. [You will need to know the formula mass of $CuSO_4 \cdot 5\,H_2O$ in order to do this calculation.]
- Using the same technique as above, prepare a second copper standard that is close to 0.2 g Cu / (25 mL H_2O). You will need to calculate the grams of $CuSO_4 \cdot 5$ H_2O that you need to add to your 25 mL volumetric flask. Have a new member of your lab team prepare this solution. Remember not to spend time getting exactly the mass of $CuSO_4 \cdot 5\,H_2O$ you calculated. Get it close but record the exact mass used (ex. 0.488 g $CuSO_4 \cdot 5\,H_2O$). Calculate the exact number of grams Cu in your 25 mL solution.
- Finally prepare your third standard that is around 0.4 g Cu / (25 mL H_2O).
- Prepare a table in your notebook that lists the following information:
  - Sample number
  - Mass of $CuSO_4 \cdot 5\,H_2O$
  - Mass of copper ions in 25 mL solution
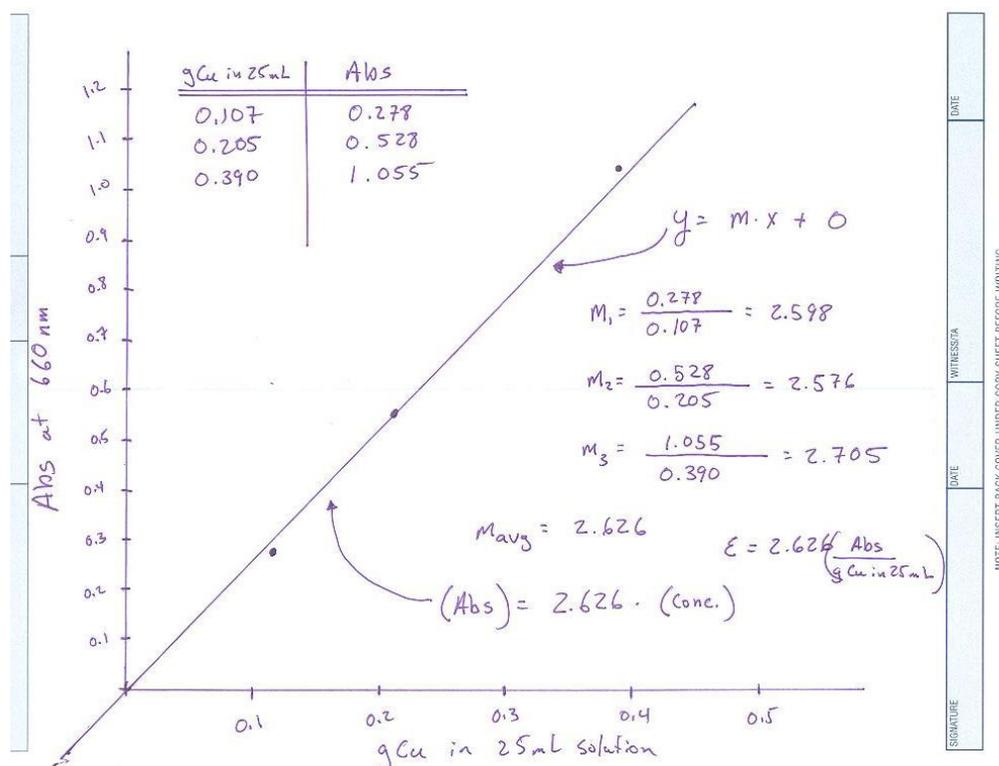 - Absorbance

Your lab bench should now have four volumetric flasks containing copper solutions of varying copper concentration. Three of the solutions are of known copper concentration (standards) and two of unknown concentration. Can you tell by eye between which two standards your unknown sample falls in concentration? Our eyes are pretty good at determining relative concentration (qualitative), but we have a spectrometer that can do a comparison quantitatively. Refer to the appendix for how to set-up and run your spectrometer

- Set the spectrometer wavelength to the value you determined best for this experiment (wavelength of maximum absorbance)
- Record the absorbance for all solutions. Be sure that all members of your group read the absorbance on each sample. Analog machines, like Spectronic 20s, can be tricky to read. It is frequently easier to record the % transmittance (%T), which is a linear scale on those instruments, in addition to the absorbance. It is worth mentioning that the higher the absorbance, the lower the transmittance value. This is logical since the light that is transmitted is simply the light that is not absorbed by a sample. This number is much easier to read on the analog spectrometers and can be converted to absorbance using the formula:

$$Abs = -\log(T) \qquad T = \%T / 100$$

- The absorbance data that you collect should be recorded in a well formatted table in your laboratory notebook. This will be an important component of a good laboratory notebook. Fortunately our colorimeter will measure absorbance directly.

From the absorbance data, a calibration curve of the standard solution will be generated. The calibration line will be the result of plotting Absorbance at 660 nm vs. copper concentration (g Cu/ 25 mL H_2O). See the example below.

The linear plot can be fit to the Beer-Lambert formula:

$$Abs = (\varepsilon)(l)(conc.) + 0.00$$

$$y \ = \ m \cdot \ x \ + \ b$$

The slope of the line, ε, is called the molar absorptivity. The path length of the sample (l) is constant in this experiment (1cm), but is important in determining sample absorbance.

The intercept of the plot is zero and should be thought of as zero absorbance when there are no copper ions present.

- From your data determine the value of ε by determining slope (rise over run) for your three data points. You will have three separate determinations of the slope. Average these three slopes to obtain ε. Alternatively, a linear regression function can be employed to determine the slope using a spreadsheet program such as Excel.
- From the absorbance of the unknown copper solution and the formula just determined from the average slope, calculate the amount of copper in your 25 mL solution.
- Calculate the percent copper in the $Cu_XCl_Y \cdot ZH_2O$ sample.
- You should now have the % H_2O and the % Cu present in $Cu_XCl_Y \cdot ZH_2O$. Determine the % chloride in the compound by subtraction.
- Be sure that the hot plates and spectrometers at your desk are off. Place the copper solutions in the aqueous waste beaker in the hood. Rinse clean all volumetric glassware and Spec 20 cuvettes. Return this glassware to your TA or to the oven in the lab.

Determining Molecular Formula:

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 in the sample. The problem with 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 water molecules 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. $CrCl_3 \bullet 6H_2O$).

To obtain the atom ratios (chemical formula) from the mass percent, you need to divide each percentage by the weight of each atom. 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.

$$\frac{48.1 \cdot g \cdot Ni}{58.7 \cdot \frac{(g \cdot Ni)}{mol}} = 0.819 \, mol \, Ni \qquad \frac{51.9 \, g \cdot PO_4}{95 \cdot \frac{(g \cdot PO_4)}{mol}} = 0.546 \, mol \, PO_4$$

It is interesting that there is actually more nickel in the sample than phosphate. The atomic composition of this compound is then $Ni_{0.819}(PO_4)_{0.546}$. This non-integer representation of the atom ratios is exceedingly ugly and should be converted to whole numbers by dividing each atom amount by the smallest atom amount.

$$Ni_{\frac{0.819}{0.546}}(PO_4)_{\frac{0.546}{0.546}} = Ni_{1.5}(PO_4)_1$$

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We still find that atom quantities are not expressed as integer values. 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.

Convert your mass percents for Cu, Cl and H_2O into atom ratios by dividing the percentage by the molar mass of the respective atom or molecule. Report the whole number ratios in your notebook.

Be sure to turn in your duplicate notebook pages.