Determination of an Unknown Metal Sulfate Hydrate

CHEM 210 Prelab Notes

Overview of MSH determination

- A metal sulfate hydrate (MSH) is an ionic compound with the general formula: $\mathbf{M_x(SO_4)_y \cdot z\ H_2O}$
- The compounds are solids in crystalline form. At the particle level, they consist of a repeating lattice of metal and sulfate ions, with water molecules incorporated into the crystal in a regular pattern and in a definite ratio.
- You will be given an unknown metal sulfate hydrate sample.
- You will determine the percent water in the the structure and the percent sulfate in the structure.
- You will compare these to a set of possible unknowns to determine the unknown's identity. You may also use other qualitative observations about your compound to help

Law of Definite Proportions

- This law states that in a given chemical compound, the mass percentages of elements in a compound are always the same.
- If the mass percent of elements individual elements are the same, we could conclude that the mass percent of the same groups of atoms would also be the same.
- We will exploit this property by determining the mass percent of H_2O molecules and the mass percent of SO_4^{2-} ions in our MSH unknowns.
- Then, we will compare those values to a set of possible unknowns. Because the mass percentages are a constant for a given compound, one unknown should closely match both percentages.
- The law of definite proportions can be extended to the particulate level of matter to conclude that a given compound will always have the chemical formula.

Calculations with the Possible Unknowns

- There are a number of possible MSH that your unkown could be. In order to make the necessary comparisons, you will need to calculate the molar mass and the % water and % sulfate in each.
- Consider the possible unknown: $\mathbf{NiSO_4 \cdot 6\ H_2O}$
  - *Calculate the molar mass of the compound.*
  - *Calculate the % H_2O in the compound.*
  - *Calculate the % SO_4^{2-} in the compound.*

Dehydration of the Unknown

- You will not know the formula of your MSH.
- In order to determine the mass percent of water, you must determine the mass of water (part) in a sample of known mass of the MSH (whole).
- With heating, the water in most metal sulfate hydrates will be released from the crystal structure as water vapor. This process is called dehydration.
- The following is a balanced reaction for the dehydration of nickel (II) sulfate hexahydrate:

$$\mathbf{NiSO_4 \cdot 6H_2O_{(s)} \rightarrow NiSO_{4\,(s)} + 6H_2O_{(g)}}$$

- Note that in the product side, only the metal sulfate remains as a solid. The H_2O has left as a vapor (gas).
  - *On the reactant side, the water is part of the compound. On the product side, it is completely separate.*

Dehydration of the Unknown

- To determine the % water in a known sample of the MSH, we will dehydrate the sample by heating.
- Based on the data collected, we will then calculate the mass percent of water using the equation:

$$\text{mass \% } H_2O = \frac{\text{mass of } H_2O}{\text{mass of MSH sample}} \times 100\%$$

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Part 2

Summary of Part 1/ Intro to part 2

- In part 1 of the experiment, you will determine the % H_2O in your compound.
- Based on that value, you should be able to make a preliminary determination of the identity of your unknown, or narrow it to a few choices.
- In part 2, you will determine the % SO_4^{2-} in your compound.
- With both pieces of data, you should be able to make a fairly certain identification of your unknown.
- You will then confirm physical properties like color, or melting point range with the literature to see if that assignment makes sense.

% Sulfate Determination

- Sulfate cannot be driven off from the crystal structure in the same way water was.
- Instead, the sulfate ion in your unknown will be isolated through a precipitation reaction with barium chloride.
- To do this, you will first dissolve a massed sample of you MSH in a water. When it dissolves, the sulfate ion is solvated by water molecules, and will be free in the solution.
- *Consider what happens to $NiSO_4 \cdot 6\ H_2O$ when it is dissolved in water:*
- All of the MSH's dissolve in an analogous process.

% Sulfate Determination

- Once dissolved in solution, the SO_4^{2-} can be precipitated as $BaSO_4$ by adding $BaCl_2$ solution to it.
  - Note: The $BaCl_2$ is also dissociated in solution:
- The dissolved sulfate ions and the barium ions are attracted to each other and form a solid when the two solutions are mixed:
- *This reaction is the same, regardless of your unknown, as all unknowns contain sulfate ions.*

% Sulfate Determination

- The crystals of the precipitate that form are very small.
- The crystals must be ***digested*** by heating them.
  - In the digestion process, smaller crystals dissolve more rapidly than larger crystals.
  - Larger crystals have a larger surface area for the continuing recrystallization of the $BaSO_4$.
- Once digested, the crystals are filtered, dried, and massed.
- The mass of the $BaSO_4$ can then be used to calculate the mass of just the sulfate ion (all of which came from your unknown).
- The percent sulfate can then be calculated.

Determining the volume of $BaCl_2$ solution to use.

- An excess of $BaCl_2$ must be used to ensure all of the sulfate ion from the unknown is precipitated.