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: $\text{M}_x(\text{SO}_4)_2 \cdot z \text{H}_2\text{O}$
- 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 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 $\text{H}_2\text{O}$ molecules and the mass percent of $\text{SO}_4^{2-}$ 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: $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$
- Calculate the molar mass of the compound.
- Calculate the % $\text{H}_2\text{O}$ in the compound.
- Calculate the % $\text{SO}_4^{2-}$ 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:
  $$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}(s) \rightarrow \text{NiSO}_4(s) + 6\text{H}_2\text{O}(g)$$
- Note that in the product side, only the metal sulfate remains as a solid. The $\text{H}_2\text{O}$ 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:
  $$\text{mass % } \text{H}_2\text{O} = \frac{\text{mass of } \text{H}_2\text{O}}{\text{mass of MSH sample}} \times 100\%$$
Summary of Part 1/ Intro to part 2

- In part 1 of the experiment, you will determine the % H₂O 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 % SO₄²⁻ 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 NiSO₄•6H₂O when it is dissolved in water:
  - All of the MSH's dissolve in an analogous process.

% Sulfate Determination

- Once dissolved in solution, the SO₄²⁻ can be precipitated as BaSO₄ by adding BaCl₂ solution to it.
  - Note: The BaCl₂ 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 BaSO₄.
- Once digested, the crystals are filtered, dried, and massed.
- The mass of the BaSO₄ 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 BaCl₂ solution to use.

- An excess of BaCl₂ must be used to ensure all of the sulfate ion from the unknown is precipitated.