Determining the Formula of a Hydrate

Objectives

The purpose of this experiment is to determine the empirical formula of a hydrate. Hydrates are inorganic salts which contain a specific number of water molecules loosely attached. An example is sodium carbonate decahydrate (washing soda). The formulas for this substance is $Na_2CO_3 \cdot 10H_2O$. Not all hydrates have simple formulas like this one. For example, a hydrate of cadmium sulfate seems to have 2.66 molecules of water for each molecule of $CdSO_4$. This hydrate is best represented as $(CdSO_4)_3 \cdot 8H_2O$. When determining the formula of a hydrate you must not assume that it is one with a simple formula.

Hydrates can normally be decomposed into the anhydrous salt and water by gentle heating according to the equation below. From the data collected the number of molecules of hydrated water will be determined per molecule of anhydrous salt.

$$ZnSO_4 \cdot xH_2O \; ? \; ZnSO_4 + xH_2O$$

Procedure (Details)

You will work in pairs for this experiment.

1. Thoroughly wipe a crucible and cover with a clean cloth towel to remove dirt and other particulate matter. Then **determine the mass of the crucible (and cover) to +/- 0.01 g**.
2. Obtain about 1 g of a hydrate sample and transfer the sample to the crucible.
    a. Samples will be found on the reagent counter; be sure to write down the identification code.
    b. **After you obtain your sample, quickly replce the cap to the reagent bottle and tighten securely.**
    c. Transfer the hydrate sample to your crucible.
3. Determine the mass of the crucible, hydrate sample, and crucible cover.
4. Using your crucible tongs, place the crucible and contents back on the clay triangle. Partially cover the opening of the crucible with the cover.
5. Heat your crucible and its contents with a low flame for 5 min. Increase the flame temperature and heat with a medium flame for 5 min. Further increase the flame and heat the sample for an additional 10 min. **Do not allow the crucible to turn red. (Overheating may lead to decomposition of your sample!)**
6. **Using crucible tongs**, remove the crucible (with cover in place) from the clay triangle and place

on wire gauze on the lab bench.

7. Allow the crucible to cool to room temperature. (Hold your hand about 1 cm above the crucible to test.) Then **determine the mass of the crucible and contents (and cover) to +/- 0.01 g**.
8. Be sure to note any changes in appearance of your sample in your lab notebook.
9. Heat your sample to constant mass.
    - Reheat your crucible, with the cover ajar as before, for 10 min. using a hot flame. Allow the crucible to cool to room temperature and reweigh.
    - The difference between the masses of the crucible plus sample, after the first and second heating, should be no greater than 0.03 g.
    - If the difference is greater than 0.03 g, repeat the heating and cooling procedure until the difference between consecutive heatings is less than this limit.
    - You will then have heated your sample to a "**constant mass**".
10. After you have heated your crucible and contents to constant mass, transfer the crucible's contents to the disposal container.
11. You may wish, if time permits, to perform a second determination.
12. Determine the formula of the hydrate. The possible hydrates are: $ZnSO_4 \cdot xH_2O$, $CaSO_4 \cdot xH_2O$, $MgSO_4 \cdot xH_2O$