Hydrates

Background

It is common for salts (ionic compounds) to be hydrated; that is, to have specific amounts of water bonded to the ions in the salt. This water is called water of hydration or water of crystallization. Some examples of hydrated salts are: $CaCl_2 \cdot 2H_2O$, $Fe(NO_3) \cdot 9H_2O$, $MgCO_3 \cdot 3H_2O$, $Na_2SO_4 \cdot 10H_2O$. In the formula a dot precedes the number of moles of water per mole of anhydrous (without water) compound. The water molecules are usually not strongly held and often can be removed by heating.

$$BaCl_2 \cdot 2\ H_2O \xrightarrow{heat} BaCl_2 + 2H_2O$$

If a weighed hydrate sample is heated and then weighed again, the mass of water released can be determined and the percent water calculated. For example if a 10.00 g sample of a hydrate is found to have a mass of 8.53 g after heating, then the mass of water released can be calculated as follows:

$$10.00\ g - 8.53\ g = 1.47\ g$$

and the percent water is:

$$\frac{1.47\ g}{10.00\ g} = 14.7\ \%$$

This experiment is in two parts. In the first part you will verify that when a hydrate is heated, water is produced. The presence of water can be detected by using paper saturated with anhydrous cobalt (II) chloride. $CoCl_2$, which is blue, reacts with water to form red $CoCl_2 \cdot 6H_2O$.

Equipment

From the common drawer:

- ring stand and ring
- wire gauze
- bunsen burner
- utility clamp

From your drawer:

- large test tube
- medium test tube
- crucible and lid
- 250 mL Erlenmeyer flask
- watch glass

Procedure

Carefully put about 4 or 5 g of $CuSO_4 \cdot 5H_2O$ into a clean, dry large test tube so that none adheres to the sides of the test tube. Clamp the test tube to a ring stand so that the mouth is pointed slightly downward (not at so steep an angle that the copper sulfate falls out!). Place a clean, dry medium test tube upright in a flask so that its mouth is under the mouth of the test tube containing the hydrate. Heat the hydrate gently at first and then gradually increase the heat until the blue hydrate becomes white, collecting the liquid produced in the test tube in the flask. (If it is heated too strongly, it will decompose yielding black copper (II) oxide.) Now gently heat the entire test tube to vaporize any liquid that has condensed on its walls. Avoid directly heating the clamp as it may cause it to melt. Describe the odor and appearance of the liquid collected. Place a drop of the liquid on a piece of blue cobalt chloride test paper. Also place a drop of deionized water on the test paper. Record your observations. Divide the white residue from the test tube into two portions on a watch glass. Put a drop of the liquid collected on one portion and a drop of deionized water on the other. Record your observations. What do your observations tell you about the nature of the liquid collected by heating the hydrated copper (II) sulfate? Why?

Wash and rinse a crucible and lid. Place them on a clay triangle on a ring stand and heat them until they are dry. Allow them to cool to room temperature, weigh them and record the mass. Obtain an unknown hydrate from your instructor and record its number. Put about 2 or 3 g of the unknown in the crucible; weigh the crucible, lid, and contents and record the mass. Put the crucible on the clay triangle with the lid slightly ajar so that water vapor can escape. Heat the crucible and contents gently for about 5 minutes and then more strongly for 10 – 15 minutes longer. Close the cover of the crucible and allow it to cool to room temperature on the clay triangle. **Do NOT set the hot crucible on a cool contertop–it will break!** When it has come to room temperature, weigh it and record the mass. Heat the crucible again for about 5 minutes, allow it to cool, weigh it, and record the mass. If the two weighings do not agree within 0.05 g, heat, cool, and weigh the crucible, lid, and contents again. Continue this process until the masses do agree.

Calculate the percent water in your unknown hydrate

Dispose of all solid waste in inorganic solid waste container. If some solid gets stuck, rinse with deionized water into aqueous metal waste container.