

Gravimetric Determination of the Water of Hydration of Epsom Salt

Objective: To determine the relative hydration of a sample of Epsom salt

Concept to be Tested: The quantity of a component present in a mixture can be determined by gravimetric analysis.

Textbook References: McMurray and Fay: Chapters 1.4-1.6, and 3.3-3.7

Techniques: Review *General Laboratory Procedure*

Introduction

Gravimetric analysis, by definition, includes all methods of analysis in which the final stage of the analysis involves determining the amount of material present by use of the analytical balance. In the experiment today, the compound decomposes to liberate a volatile product. The difference in weight corresponds to the gain or loss of a specific substance from the sample. This corresponds to a molar quantity of material that was gained or lost. This change can be used to determine the percent composition or moles of a compound that are associated with that particular sample. This is one of the simplest types of gravimetric analysis.

For this specific experiment, you will be dehydrating a sample of magnesium sulfate hydrate, Epsom salt, $\text{MgSO}_4 \cdot \text{XH}_2\text{O}$. The $\cdot \text{XH}_2\text{O}$ means that the crystalline form of magnesium sulfate that you started with contains an unknown number of moles of water per mole of magnesium sulfate. Typically, Epsom salt has the formula $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$. This varies over the year to between 1 and 9 molecules of water depending upon the relative humidity and the conditions under which the compound is stored. This is a stable compound and regardless of where you find this particular substance, it will always have water associated with the crystal. Since the water has not technically reacted with the magnesium but is part of the particular crystal structure, it is possible to remove the water from the sample without altering or decomposing the MgSO_4 . You will do this by heating the $\text{MgSO}_4 \cdot \text{XH}_2\text{O}$ to dissociate the water from the crystal and creating the anhydrous form of the chemical, MgSO_4 . This is what you will do in today's experiment.

In this analysis, a weighed sample of starting material will be heated to drive off the volatile component. The residue is weighed after heating and the weight of the volatile component is determined by difference. By knowing both the identity of the volatile component and the identity and chemical composition of the residue, it will be possible to determine the relative ratio of the two components in the starting material.

Epsom salt or Epsom salts get their name from a mineral bath in Epsom, England. Epsom salts have been used medicinally for over 2000 years. The residue from today's experiment may be safely disposed of by cleaning your glassware with water and flushing the solution down the drain.

Example: Calculate the number of waters of hydration present in a sample of Epsom salt given the following data. A 1.5000 g sample is heated with stirring until the crystals are reduced to a dry powder. After cooling, the residue was found to weigh 0.8746 g.

The difference in weight of the sample, 1.5000 g, and the residue, 0.8746 g, is 0.6254 g. Therefore the initial sample contained 0.6254 g of H₂O and 0.8746 g of MgSO₄. To determine the formula of the starting material it will be necessary to determine the moles of water and moles of MgSO₄ present in the sample. The ratio of these two gives the formula of the starting material.

$$\text{moles H}_2\text{O} = \frac{0.6254 \text{ g}}{18.01 \text{ f.wt. H}_2\text{O}} = 0.03473 \text{ moles}$$

$$\text{moles MgSO}_4 = \frac{0.8746 \text{ g}}{120.36 \text{ f.wt. MgSO}_4} = 0.007266 \text{ moles}$$

$$\text{ratio H}_2\text{O to MgSO}_4 = \frac{0.03473 \text{ mol H}_2\text{O}}{0.007266 \text{ mol MgSO}_4} = 4.780$$

Rounding the answer to the nearest whole number, there are an average of 5 water moles of water per formula unit of MgSO₄ or a formula of MgSO₄·5 H₂O.

General Experimental Information

It is important to avoid careless errors when performing gravimetric analysis. Perhaps the most common is the use of dirty glassware. Make certain that the beaker you use is clean and dry. While it is not necessary to flame dry the beaker when using Epsom salt, there must not be any visible droplets of water present in the beaker. You must carefully check the beaker for the presence of small cracks or stars. A crack or star will expand when the beaker is heated causing the beaker to shatter. In addition to the obvious safety hazard, this also means that you will have to start over with the experiment.

When you first begin heating the beaker with a Bunsen burner you will notice condensation appear on the **outside** of the beaker. This is water vapor that was produced by combustion of the gas that is condensing on the relatively cool walls of the beaker. This disappears as the beaker is heated further. As the beaker becomes hot, you will then notice water forming on the **inside** of the beaker. This is water that is coming from your sample. This will also evaporate as the beaker becomes still hotter. You must make certain that there are no visible drops of water **inside** the beaker *before* you finish heating.

After heating, you will have to allow the beaker and its contents to cool to near room temperature. A hot beaker does not have the same mass as a cold beaker, but the air currents that are set up by the heat radiating from the hot beaker will make it very difficult to obtain an accurate weight. Patience is imperative.

Murphy's First Law of the Laboratory: Hot glass looks exactly the same as cold glass.
First Corollary: Hot metal looks exactly the same as cold metal.

Experimental Procedure

This experiment can be performed using either a Bunsen burner or a hot plate. Your instructor will tell you whether to follow the Bunsen Burner or Hot Plate Procedure to remove the water from your sample of Epsom salt. Regardless of the procedure you use, the subsequent cooling and weighing steps after you have obtained a dry powder are identical.

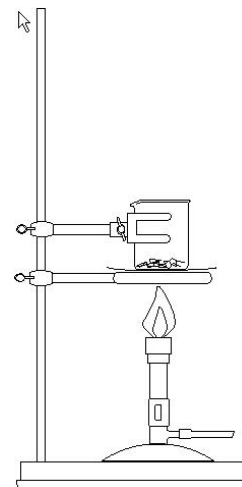
Obtain a clean, dry 100 mL beaker. Make certain that the beaker does not have a crack or star in it. Weigh the beaker to the nearest 0.0001 g and record this weight. Then add between 1 and 2 g of Epsom salt to the beaker. Determine the weight of your sample to the nearest 0.0001 g and record this weight.

Bunsen Burner Procedure

Set up a ring stand with a ring clamp, wire screen, and second clamp. Estimate the height of the Bunsen burner flame and adjust the ring clamp so that the flame will just touch the wire screen.

Important! Do not use the hottest portion of the flame. Once you have positioned this clamp, place the beaker onto the wire gauze and then securely clamp the beaker.

Place the Bunsen burner under the beaker and begin stirring the contents of the beaker with a clean glass rod. Continue stirring until the contents are reduced to a fine powder. This should take approximately 3 minutes. **Do not continue heating after a dry powder is obtained.**



Hot Plate Procedure

Obtain a hot plate from the storage cabinet. Place your beaker onto the hot plate and turn on the heating controls. You will need to use a medium setting for the heat control when you begin. You may need to adjust the heat setting either up or down after the plate gets hot. Stir the contents of the beaker with a clean glass rod. If the crystals do not appear to melt or bubble within 5 minutes, you will need to turn up the heat setting. Continue gentle stirring until a dry powder is obtained. This will take approximately 3 minutes. **Do not continue heating after a dry powder is obtained.**

After a dry powder is obtained.....

Allow the beaker to cool and weigh the beaker and residue to the nearest 0.0001 g. You will use this data to calculate the number of moles of water present per formula weight unit of MgSO_4 .

You will repeat this procedure with a second sample. (You can save time by beginning the second trial before the first beaker has cooled enough to be weighed.) Use a fresh dry 100 mL beaker for this second trial. You will average your results to determine the average number of moles of water present in your MgSO_4 sample.

Cleanup

Disassemble your set-up and replace the items in the storage cabinets where you obtained them. Clean the glassware with water. It is safe to dispose of the dried magnesium sulfate in the trash can. The residual magnesium sulfate will dissolve in water and can be disposed of down the drain. Return the beakers and stirring rod to their appropriate storage areas within the laboratory.

Safety

You must wear departmentally approved eye protection at all times while you are in the laboratory. Bunsen burners will be in use in the laboratory. Flames, hot glassware and metal can cause burns unless due caution is exercised. Carefully check the glassware for the presence of small cracks or 'stars' which can cause the glassware to break (or shatter) when heated.

Respiratory Distress Warning! Over heating the dried magnesium sulfate can cause it to further decompose and liberate SO_3 that can cause respiratory distress. If you suffer from asthma or other breathing difficulties, be aware of this. If you experience any difficulty in breathing notify your instructor and leave the laboratory immediately.