Determination of Water in a Solid

Objective

You will use techniques of quantitative analysis to determine the percent by mass of water in a solid.

Introduction

The accurate determination of the composition of substances is essential in modern technology and important in our everyday life. Typical problems in which analytical chemistry plays a role are determining the compositions of lunar and other extraterrestrial samples, monitoring harmful levels of pollutants in the atmosphere above our cities and in our water supplies, maintaining quality control over drug and food products, detecting trace impurities in ultra pure semi-conductor materials (transistors, diodes, etc.), and making clinical investigations which determine the nature and concentration of materials in biological fluids.

As can be seen from the examples given, analytical determinations may be either qualitative or quantitative in nature. A qualitative analysis is aimed at determining the identity of a substance (what is present); a quantitative analysis determines the amounts of known substances present in a particular sample (how much is present).

This experiment will introduce you to precision weighing and quantitative analysis techniques. You will determine the percent by mass of water in a sample of solid. The solid contains one or more hydrates – salts that crystallize with a definite number of water molecules in each formula unit. For instance, barium chloride dihydrate, BaCl₂·2H₂O, is a hydrate containing two molecules of water (water of crystallization) for each BaCl₂ unit. You will heat the solid to drive off the water of crystallization, and from the loss in mass, calculate the percent of water that was present.
**Pre Lab Problem (answer on separate paper)**

A student placed a solid hydrate in a crucible and heated it to drive off the water of crystallization. The following data was collected:

<table>
<thead>
<tr>
<th>Description</th>
<th>Mass (g)</th>
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<tbody>
<tr>
<td>Mass of empty crucible + cover</td>
<td>18.346</td>
</tr>
<tr>
<td>Mass of crucible + cover + sample:</td>
<td></td>
</tr>
<tr>
<td>before heating</td>
<td>20.652</td>
</tr>
<tr>
<td>after heating</td>
<td>19.617</td>
</tr>
</tbody>
</table>

1. What is the mass of the hydrate?
2. What is the mass of the water driven off?
3. Calculate the percent by mass of water in the hydrate.

**Equipment & Reagents**

- Crucible and cover
- Triangle
- Crucible tongs
- Bunsen burner
- Ring stand
- Ceramic tile(s)
- Iron ring
- Stainless steel spatula or scoop
- Unknown hydrate

**Procedure**

**CAUTION:** The solids may be poisonous. Wash skin contact areas profusely.

1. Check your crucible for any hairline cracks, and replace if necessary. The sound when you tap it should be a “ping,” not a “thud.” Practice using your crucible tongs to place the cover on the crucible and to pick up the covered crucible. Your instructor will demonstrate the proper use of crucible tongs.

2. Clean and dry the crucible. Place the empty crucible with cover ajar on a triangle, as shown in the figures below. Heat gently for a few minutes with a Bunsen burner. Adjust the burner for a hot, blue flame; the inner blue cone should come about 2-3 cm above the burner. Heat the crucible strongly for five minutes, with the crucible about 2 cm above the inner blue cone. The bottom of the crucible may glow a dull red during this time.
LAB TWO

CAUTION: After the crucible stops glowing it may still be very hot!

3. Turn off the burner. With crucible tongs, place the cover on the crucible. Use the tongs to move the covered crucible to cool on a clean, dry ceramic tile placed on the lab bench. While transporting the crucible with the tongs, hold a ceramic tile just underneath the bottom of the crucible so that it won’t shatter if dropped.

NOTE: Your instructor will demonstrate the proper way to determine whether the crucible and cover have reached room temperature. If the crucible and cover are weighed when they are warmer than room temperature, an error will result from buoyancy; the apparent mass will be low.

As always, don’t forget to make sure the balance reads “zero.” Be certain that your fingers are clean and dry when you handle the crucible and cover so that oil and moisture from your fingers will not affect the mass.

4. When the crucible and cover are fully cooled, measure and record their combined mass to the nearest milligram (0.001 g).

5. Obtain an unknown solid and record the code. Using a stainless steel scoop or spatula, add to the crucible between 2.2 and 2.5 grams of the solid. Remember that you should never add chemicals to a container while it is on the balance! Replace the cover, weigh, and record the mass of the combined crucible, cover, and sample to the nearest milligram.

6. Again place the crucible in the triangle with the cover ajar. Heat it gently for a few minutes and then, using a hot, blue flame, heat it for 15 minutes. Turn off the burner, and, as before, use tongs to cover the crucible and move it (over a tile) to the ceramic tile to cool. Allow the crucible and contents to reach room temperature. Measure and record the mass of the crucible, cover, and residue to the nearest milligram.

7. Repeat Step 6 by heating, cooling, and reweighing. Compare the mass with the mass obtained after the first heating of the sample. If necessary, repeat Step 6 again until two successive weighings agree within 20 milligrams. This procedure is called heating to constant weight. Consider the last weighing to be your final weight.

8. Cleanup

Soak the crucible in tap water to remove the residue. Use dilute hydrochloric acid, if necessary, to remove any residue that remains. Rinse the cleaned crucible with distilled water before setting it to dry beside the sink. Clean your lab area before being signed out by your lab assistant.
Report: Determination of Water in a Solid

Data

Unknown Code ____________

Mass of empty crucible & cover ____________ g

Mass of crucible & cover & solid:
  before heating ____________ g
  after heating ____________ g
  after 2nd heating ____________ g
  after 3rd heating ____________ g

Results

Mass of sample ____________ g
Mass of water in solid ____________ g
Percent water by mass ____________ %

NOTE: Percent water by mass can be found by using the equation:

\[
\left( \frac{\text{Mass of water}}{\text{Mass of sample}} \right) \times 100 \%
\]

Calculations:
Post Lab Questions

1. While carrying the crucible and dried sample to the balance for final weighing in the procedure, a student unknowingly spilled some of the sample out of the crucible. What effect will this have on the student’s calculated value for percent water? Will it make the value too high or too low? Explain.

2. In the experiment, you were instructed to allow your sample and crucible to cool to room temperature before you completed your final weighing. A conscientious student allowed the crucible to cool for a full week with the cover ajar so that the sample inside was exposed to the air. Can you identify any problems which might be caused by this change in procedure? Would the apparent percent water be too high or too low? Explain