Experiment 2B

OBTAINING WATER FROM A MARTIAN ROCK

MATERIALS: Small test tube (20 x 150 mm), 2 porcelain crucibles, small clay triangle, crucible tongs, desiccooler, Bunsen burner, striker, ring stands, iron ring, clamp, 100 mL beaker, weighing boat, disposable plastic dropper, CuSO₄·5H₂O, unknown hydrate.

PURPOSE: The purposes of this experiment are: (1) to study the properties of a hydrate; (2) to determine the percent by mass of water in a hydrate; and (3) to determine the feasibility of hydrates as possible water sources.

LEARNING OBJECTIVES: By the end of this experiment, the student should be able to demonstrate the following proficiencies:

1. Explain why crucibles are heated before an initial mass determination and why heated crucibles are cooled in a desiccooler.
2. Explain what a hydrate is and give examples of hydrates.
3. Calculate the percent by mass of water in an ionic solid hydrate.

PRE-LAB: Complete the pre-lab questions on page E2B-5 before lab.

DISCUSSION:

Ever since people have gazed into the night sky, there has been a fascination with outer space. In the 1870s, Italian astronomer Giovanni Schiaparelli reported seeing “channels” on Mars with his telescope, and soon began the questions about life on other planets. Next to Earth, Mars has the most hospitable climate in our solar system. Did it once support life? Could it support human exploration or colonization?

The environment on Mars is harsh, but not unlike Earth’s climate billions of years ago. Mars lacks a UV-protecting ozone layer, its thin atmosphere consists mainly of CO₂ (95%), and the average surface temperature is about -81°F (-63°C) ranging from 75°F to -100°F. Also, at present, there is no evidence of liquid water on Mars, a necessary component for life (as we know it). However, despite its reputation as a dry planet, Mars does contain water, in the form of ice in its polar caps (beneath frozen CO₂), possibly underground, and trapped in mineral deposits called hydrates. In fact, these hydrates support claims that water was once abundant on Mars. The theory suggests that large bodies of water evaporated over a long period of time with most of the water becoming trapped within the structures of these ionic salts. NASA’s rover Opportunity recently revealed evidence of a hydrated iron sulfate mineral called jarosite from a rock dubbed “El Capitan” in the crater at Meridiani Planum. Professor Steven Squyres from Cornell reported that “We have concluded that the rocks here were once soaked in liquid water. It changed their texture, and it changed their chemistry. We believe that this place on Mars for some period in time was habitable. It was a ground-water environment, the kind of place that would have been suitable for life. That doesn’t mean life was there. We don’t know that. But this was a habitable place on Mars at one point in time.”

Other minerals found on Mars (by NASA) include hydrates of magnesium sulfate and calcium sulfate as well as hematite (iron oxide) which gives Mars its red appearance and nickname as the Red Planet. While these hydrates may provide evidence of the existence of water on Mars, their presence may also prove to be very beneficial for future human exploration of the planet. These hydrates represent stored water that could be accessed to support life on Mars. One source claims that the cost for sending a gallon of water to the moon is $250,000. Expecting a spacecraft to carry enough water to support a crew would be unrealistic, so viable ways of producing one’s own water or extracting water from the environment must be found in order to make space exploration or colonization a reality.

In this experiment, you will study the properties of a hydrate and determine the water content of an unknown hydrate. The unknown hydrates include some which have been discovered on Mars:

- MgSO₄·7H₂O Episome
- CaSO₄·2H₂O Gypsum
- Li₂SO₄·H₂O evidence of other Li compounds found on Mars

¹Cornell press release on 2 March 2004 at http://www.news.cornell.edu/releases/rover/Mars.jarosite.html
A hydrate is a chemical compound that contains a specific integer number of moles of water relative to each mole of the primary ionic solid. The water molecules are usually held loosely within the crystal. Moderate heating of the hydrate will drive off the water, leaving the anhydrous, or water-free, ionic solid.

The chemical formula for a hydrate is written in a special form, using a dot between the formulas for the ionic solid and the water. When naming hydrates, the ionic solid is named following standard rules of nomenclature. The number of water molecules is indicated using the Greek prefix (1 = mono; 2 = di; 3 = tri; 4 = tetra; 5 = penta; 6 = hexa; 7 = hepta; 8 = octa; 9 = nona; 10 = deca) with the water portion of the compound referred to as "hydrate". For example, CuSO₄·5H₂O is named copper(II) sulfate pentahydrate.

PROCEDURE:

Work with a partner to collect data for this experiment. Set up 2 ring stands to heat 2 crucibles simultaneously.

Part A. Crucible Preparation

1. Observe the correct usage of the Bunsen burner, as demonstrated by your instructor. Make any special notes for future reference.

2. Determine a method for identifying each crucible so that you can distinguish between them. Do not use a grease pencil to mark the crucibles since markings will melt away during heating. If a crucible is broken or cracked, obtain another one.

3. The first step in this procedure involves heating the empty crucibles so that they are completely dry and free of all combustible materials such as fingerprint grease. Attach an iron ring to a ring stand. Place a small clay triangle in the center of the iron ring. Position a clean crucible on the clay triangle as in Figure 1. There should be enough equipment so that both of the crucibles can be heated simultaneously on 2 separate ring stands.

4. With a Bunsen burner, heat each crucible for at least five minutes using a hot flame that has been adjusted so that the tip of the inner cone is just below the crucible. To ensure adequate heating, the bottom of the crucible and clay triangle should have an orange-red glow.

5. Allow each crucible to cool for one minute on the clay triangle before placing it into the desiccator using the crucible tongs provided. Do not place hot crucibles on the bench top or on paper. This will cause them to pick up dirt and other contaminants (and possibly ignite the surface).

6. Immediately replace the lid of the desiccator and allow the crucibles to cool to room temperature. This will take as long as or longer than the five minute heating period. The dry environment within the covered desiccator will enable the crucibles to cool without absorbing water from the air. Keep the lid on the desiccator whenever possible to extend the life of the desiccant.

7. For the rest of the experiment, the crucibles should ONLY be handled with tongs. While your crucibles are cooling, start work on Part B.

8. When the crucibles have cooled to room temperature, determine the mass of each with an analytical balance. Make sure you know which mass corresponds to which crucible.
Part B. Studying the Properties of a Known Hydrate

1. Place about 0.4 g of CuSO₄·5H₂O in a small test tube. Attach it to a ring stand with a clamp at an angle of about 45° for this experiment (see Figure 2). Be careful with the fragile test tube; it will break if too much pressure is applied. Attach the clamp near the top of the test tube so that it won’t melt when the tube is heated. Point the test tube away from people!

2. With a Bunsen burner flame, gently heat the contents of the test tube while continuously moving the flame. What do you notice with time and heating? Do you observe water vapor condensing near the top of the test tube? Why does this occur? Where did the water come from? Record your observations.

3. After the solid has converted to its anhydrous form, allow it to cool to room temperature. How do you know that a change occurred to the hydrate?

4. Note the temperature of the cooled test tube near the solid. Add a few drops of water to the tube. Feel the test tube now. What do you notice? Does this process give off heat or absorb it? Mix the contents of the tube gently to help dissolve some of the solid, adding a few more drops of water if necessary. Not all of the solid will dissolve.

5. Heat the solution slowly. The liquid will boil vigorously if heated too strongly, especially as the water escapes. Continuously move the flame. As the water is driven off, you may notice the original hydrate reform, and then convert to its anhydrous form as more heat is added. Stop heating when any solid forms. Further heating may result in conversion of the anhydrous form to yet another decomposition product. From these tests, you should be able to imagine how the hydrates on Mars formed over time and how “rocks” can contain water.

6. When it cools, put the used test tube with its contents in the designated waste container. Finish the last step of Part A and continue with Part C.

Part C. Unknown Hydrate

Scenario:
The NASA rover Spirit has collected soil samples from various locations on Mars. These locations were chosen because they are possible sites where water once existed, are accessible by the rover, and are potential regions for future human exploration or colonization. The samples have been pre-analyzed and determined to be essentially a single hydrate material. Your group will be given one of the hydrate unknowns and your job is to determine its water content, identify the hydrate, and decide what location on Mars would be most suitable for future human inhabitants.

Unknowns:
- Martian Rock A from Utopia Planitia
- Martian Rock B from Gusev Crater
- Martian Rock C from Ares Vallis

1. Obtain an unknown hydrate (Martian rock) from your instructor. Record the sample’s unknown number.

2. Devise a procedure to determine the mass % of water in your hydrate using your pre-prepared crucibles from Part A. Do not use more than about 1.2 g for each sample. How will you know when all of the water has been removed from the hydrate? For good results, think about this important experimental consideration. Not all hydrates change color upon heating. Let your instructor check your procedure before proceeding.

3. Record your procedure clearly (as if someone else were going to follow it) and use proper technique for consistent results.
4. Calculate the mass % of water in your unknown and report your average value to your instructor.
5. Compare your results with others in the class who analyzed other unknown samples. Make sure you have the class results from all 3 hydrate unknowns (3 regions of Mars).

Clean-up:
1. The hydrate residues can be flushed down the drain with plenty of water. Wash the crucibles with water only and if necessary, use a soft brush to remove any solids. Do not use soap on the crucibles. Leave the clean, wet crucibles inverted on a piece of paper towel in the hood to dry for the next student.
2. Return all equipment to their original locations. Clean up your lab bench and balance areas.

Data Analysis:
1. From your data, calculate the average % by mass of water in your unknown hydrate with the correct number of significant figures. Identify your hydrate from the possible unknowns listed on page E2B-1. Determine the % difference (or % error) between your experimentally determined average mass % and the expected mass % of water in the hydrate.
2. Compile the class results on all 3 hydrate unknowns in a table which lists the unknown number, its location on Mars, its identity (the hydrate chemical formula), and average % of water determined experimentally.
3. Which hydrate contained the most water? Which contained the least water? Based on these results, what location on Mars would be most suitable for future human colonization?

Questions for Consideration:
1. The crucible masses in this experiment were determined using an analytical balance which measures to +/- 0.0001 g. If a top-loading balance, which measures to +/- 0.01 g, were used in this experiment instead, how would this affect the data? Comment on the accuracy and precision in the measurements. In terms of identifying the hydrates, would it matter if an analytical or top-loading balance was used? Explain.
2. When an analysis requires decomposition by heating, as the dehydration performed here, a chemist usually reheats the crucible and residue and then determines the mass again to check for a constant mass. Suppose the mass after the second heating is less than the mass after the first heating. Provide an explanation for what might cause this observation. Assume that no mistakes were made in carrying out the procedure and that all equipment was operating properly.
3. Small desiccant packages, usually containing silica gel, are added to many commercial items such as food, pharmaceuticals, electronics, and shoe boxes. Aboard ship, expensive electronic components are often stored by wrapping the item in bubble wrap or aluminum foil and packing it with a desiccant. Desiccants are also used when the military “mothballs” aircraft for extended storage, when a boiler is placed into “dry” lay-up, and when an engine component aboard ship needs to be maintained in a moisture-free environment. What is a desiccant and where was it used in this experiment? Desiccants have limited capacities but most can be reused after “reactivation”. Suggest a way to “reactivate” a spent desiccant.
4. A sustainable source of water is essential if Mars is to be inhabited in the future. Oxygen is also a requirement. Can you suggest ways of obtaining O₂ from the planet or from some other source based on your knowledge of chemistry?
5. If a human consumes about 1.9 L of water a day, how many kg of Epsomite would need to be dehydrated to support a crew of 5 for a 1 week mission?
6. For the dehydration processes studied in this lab, water was converted to its vapor form at high temperatures. Suggest a way to collect the water vapor as a liquid so that the liquid water could be stored for future use. A picture might be helpful in your description.
Complete these questions before lab.

1. Was liquid water once abundant on Mars? What evidence has been found to support this?

2. Name the following hydrates following standard chemical nomenclature rules:
   a. CuSO₄·5H₂O
   b. MgSO₄·7H₂O
   c. Li₂SO₄·H₂O
   d. CaSO₄·2H₂O
   e. What does “anhydrous” mean?

3. Based on its chemical formula, calculate the mass % of water in CuSO₄·5H₂O.

4. a. When CuSO₄·5H₂O is heated to drive off its water, the anhydrous form remains. If 1.00 g of CuSO₄·5H₂O is heated to over 150°C, how many grams of the anhydrous solid remains assuming the dehydration process is complete? How many grams of water were removed?
   
   b. Does the Law of Conservation of Mass apply for this dehydration process? Support your answer with some numbers.