Determining Solubility of an Unknown Salt at Various Temperatures

Objectives: To determine solubility of an unknown inorganic salt in water as a function of temperature; to construct a solubility-temperature curve; and identify the unknown salt by comparing experimental curve with reference curves

Materials: Unknown inorganic salts; distilled water

Equipment: 25 x 250-mm test tube with 2-hole rubber stopper; thermometer; glass or wire stirring rod; iron rings and ring stand assembly (see Figure 2); ceramic-centered gauze heating pad; 400-mL beaker; Bunsen burner

Safety: Some inorganic salts/solutions are irritants. All samples and solutions should be handled carefully. Hot glassware should be handled with care. Eye goggles should be worn at all times in the lab.

Waste Disposal: Salt solutions should be placed in the inorganic waste container provided in the fume hood.

INTRODUCTION

What do iced tea, soda pop, and saltwater have in common? These and many other substances you use every day are mixtures that chemists call solutions. A **solution** is defined as a homogenous mixture in which one or more substances dissolve completely in another substance. The substances that dissolve are called **solutes**, while the substance in which they dissolve is called the **solvent**. Take, for example, a cup of hot tea; the various compounds in the tea leaves that dissolve in the hot water are solutes, as is the sugar that is added to sweeten the tea. The hot water is the solvent. The **solubility** of a given solute is defined as the amount of solute that will dissolve in solution at a given temperature. For aqueous solutions, solubilities are typically reported in units of grams of salt per 100 mL of water (*g salt/100 mL* H_2O).

The amount of solute that will dissolve in water depends on many factors, including temperature and the nature of the solute and solvent (i.e., molecular vs ionic, polar vs non-polar). **Salts**, such as table salt (sodium chloride), are ionic compounds consisting of cations and anions; ionic compounds are usually soluble in water, but the extent of solubility will vary from one salt to another.

Generic terms, such as *concentrated* or *dilute*, can be used to describe how much solute is dissolved in solution. Chemists use the term **saturated** to describe a solution that contains the maximum mass of dissolved solute at a given temperature. Solutions that contain less than this maximum amount are called **unsaturated**, while those that contain <u>more</u> are called **supersaturated**. Supersaturated solutions are typically achieved by preparing a saturated solution at a high temperature, and then cooling the solution.

In this exercise you will examine the effect of temperature on the solubility of an unknown inorganic salt. You will observe how solubility changes with temperature and use this information to construct a solubility-temperature curve. By comparing your experimental curve with examples of known salts you will identify your unknown salt sample.

Solutions

Anyone who has made iced tea knows that the amount of sugar that dissolves in the tea changes with temperature. At high temperatures the sugar dissolves easily. As the tea cools, however, some of the dissolved sugar may recrystallize, or come out of solution. At this point, we know that the solution is saturated with respect to the solute (sugar)—the solution contains as much dissolved sugar as it can hold at that temperature, and the excess has precipitated from solution and remains undissolved.

We can prepare a saturated salt solution by mixing a measured volume of water with a greater mass of salt than can dissolve at a given temperature; excess salt remains undissolved. The system is dynamic; some salt particles may recrystallize as dissolved ions recombine to form solid, while some solid particles dissolve into solution. The total mass of dissolved salt and the overall solution composition remain constant as long as the temperature does not change.

Alternatively, we could add an excess of salt to a measured volume of water and heat the water until all the salt dissolved. At this point we have an unsaturated solution; the amount of salt dissolved in solution is less than the maximum that the water can hold at that temperature. As the water cools, the solubility of the salt decreases until the mass of salt in solution exceeds the solubility. The temperature at which salt starts to recrystallize is called the **saturation temperature**.

Effect of Temperature on Solubility

Many factors affect the solubility of a salt in aqueous solution. One of the major factors is the attractive forces between the ions in the solid salt compared to the attractive forces between the ions and the polar solvent, water. If the attractive forces between the ionic particles and the solvent are greater than the attractive forces between the cations and anions in the solid crystal lattice, then the salt will dissolve readily and heat energy will be released (i.e., the solution feels warm). Conversely, if the attractive forces between the ions is greater than the interactions between solvent and ions, then heat energy is absorbed by the solution (i.e., the solution cools down). For most salts heat is absorbed when they dissolve in water; their solubility increases as the temperature of the solution increases.

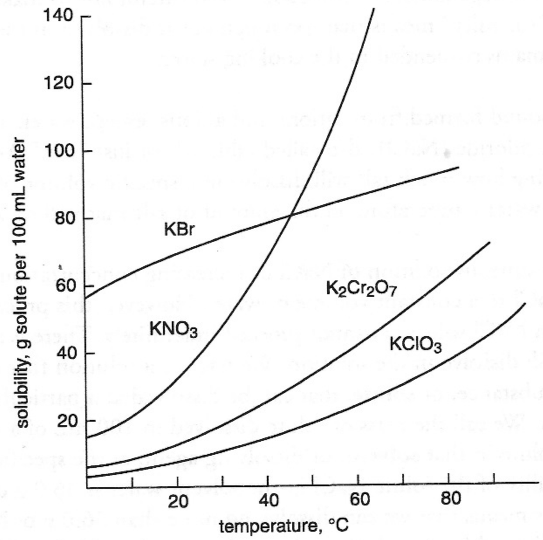

Figure 1. Solubility-temperature curves for various inorganic salts in water.

We can most easily visualize the effect of temperature on the solubility of a salt by plotting the solubility of a salt vs. saturation temperature. A **solubility-temperature** curve consists of the experimentally determined saturation temperatures on the abscissa (x-axis) and the concentration of salt in the saturated solution on the ordinate (y-axis). Solubility-temperature curves can be used to estimate the salt concentration of saturated solutions at temperatures other than those determined experimentally. In addition, unknown salts may be identified by comparing the experimental solubility-temperature curves with curves of known salts. Solubility-temperature curves of several inorganic salts are presented in Figure 1.

In this experiment you will create a solubility-temperature curve for an unknown salt by dissolving a known mass of salt in various measured volumes of water. These solutions will be heated to ensure complete dissolution of the salt. As the solution cools, you will observe the saturation temperature of each solution. The concentration of each solution can be calculated using Equation 1.

$$\text{concentration of salt solution} = \left(\frac{\text{experimental mass of salt (g)}}{\text{total volume of water (mL)}}\right) \times 100$$

Using your calculated solution concentrations and experimentally determined saturation temperatures, you will construct a solubility-temperature curve for your unknown salt and identify your unknown salt.

Pre-Lab Questions

1. What is the difference between a saturated solution and an unsaturated solution?

2. Briefly explain how the solubility of most salts is related to temperature. What general trend is observed, and why?

3. What factors determine the solubility of a salt in water? Explain.

4. A student is evaluating the solubility potassium chlorate ($KClO_3$) at various temperatures. She dissolves 2.50 g of $KClO_3$ in a total volume of 7.5 mL of water.

   a. Calculate the concentration of $KClO_3$ in this solution.

   b. The student heats the solution up to 80ºC, and then removes the test tube containing the salt solution from the hot water bath. At what temperature would you expect to see the formation of salt crystals in the solution? (Refer to Figure 1 in the Introduction.)

   c. If the solution is allowed to cool to 30ºC, what percentage of the salt remains dissolved in solution?

PROCEDURE

1. Obtain a test tube and stopper, and assemble the apparatus illustrated in Figure 2.

   NOTE: If the thermometer and stirring rod are not already inserted in the rubber stopper, insert them carefully. Apply a small amount of glycerine or other lubricant to the stopper holes. Grasp the thermometer or glass rod with a cloth or paper towel, and carefully insert into the stopper hole using a twisting motion while applying gentle pressure. Excessive force may result in breakage and possible injury. Fill the 400-mL beaker with approximately 300 mL of water, and place on the wire gauze pad as illustrated in Figure 2.

2. Place the clamp assembly holding the test tube and stopper on the ring stand, but do not lower it into the beaker at this time.

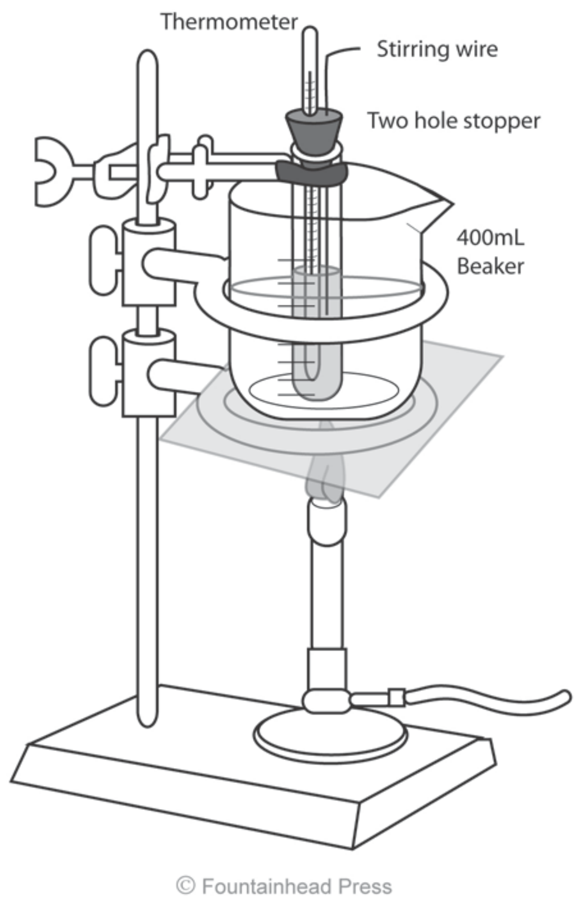

Figure 2. Apparatus for determining saturation temps of salt solutions

3. Light the Bunsen burner and place it below the wire gauze pad to begin heating the water in the beaker. Use a second thermometer to monitor the temperature of the water in the beaker. While the water is heating, prepare the original solution of your unknown salt.

I. Determining the Saturation Temperature of Original Salt Solution

4. Your TA will assign to you an unknown salt. Record the ID for your unknown on your Data Sheet.

5. Obtain approximately 5 g of your unknown salt on a piece of weighing paper or weighing boat. Weigh the sample + paper and record this mass on your Data Sheet to the nearest mg.

6. Carefully transfer <u>all</u> of your sample to your test tube. Reweigh the weighing paper or boat, and record this weight on your Data Sheet to the nearest mg.

7. Fill a clean 50-mL buret with distilled water. Dispense 3.00 mL of distilled water from the buret into the test tube containing your unknown sample. Record the actual volume of water added to your test tube to the nearest 0.01 mL.

8. Replace the rubber stopper with thermometer and stirring rod in the test tube. Be sure the thermometer bulb is about 1 cm above the bottom of the test tube.

9. When the temperature of the water in the beaker is about 80^{o}C, proceed to step 10.

10. Clamp the test tube to the ring stand using a utility clamp, and lower the test tube assembly into the hot water bath until the level of water in the test tube is aligned with the level of water in the beaker. Secure the utility clamp to the ring stand.

11. Stir the mixture in the test tube by slowly moving the stirring rod up and down in the solution. Continue mixing and heating the test tube and contents to 80^{o}C.

12. If the salt sample is not completely dissolved when the temperature of the solution has reached 80^{o}C, remove the test tube from the hot water bath and add 0.50 mL of distilled water to your test tube. Record on your data sheet the exact total volume of water added to the test tube.

13. Reheat the test tube and contents in the hot water bath to 80^{o}C while stirring carefully.

14. If necessary, repeat steps 12 and 13 until your salt sample is completely dissolved in solution at 80^{o}C.

15. After the salt has completely dissolved, loosen the clamp holding the test tube to the ring stand and lift the test tube out of the hot water bath. Reclamp the test tube in a different position so the test tube and contents are no longer immersed in the hot water bath.

16. Continue to stir the solution in the test tube while monitoring the temperature of the solution in the test tube.

17. Carefully observe the solution while it cools. Note the temperature at which the salt begins to recrystallize from solution. This is the saturation temperature for your original salt solution. Record this temperature on your Data Sheet. If you are in doubt about the saturation temperature, reheat your solution to 80 ^{o}C and repeat steps 16–18.

II. Determining the Saturation Temperature of Diluted Salt Solutions

18. Add 0.50 mL of distilled water from the buret to the solution in your test tube. As before, record the exact total volume of water added to your test tube.

19. Replace the test tube in the hot water bath and heat it while stirring until all the salt crystals have dissolved into solution.

20. Remove the test tube from the hot water bath and allow the solution to cool while stirring. Note the temperature at which salt crystals begin to form. Record the saturation temperature for your diluted salt solution on your Data Sheet.

21. Repeat Steps 18–20. If the saturation temperature does not change significantly with 0.50 mL additions of distilled water, add 1.00 mL increments.

22. If the saturation temperature falls below room temperature, replace the hot water bath with an ice bath. Add several more 0.50 or 1.00 mL increments of distilled water and repeat steps 18–20 until you have obtained saturation temperatures for at least eight diluted salt solutions.

Wash hands thoroughly with soap or detergent before leaving the lab.

CALCULATIONS

1. Calculate the solubility of your unknown salt (in grams of salt per 100 mL of water) at the original saturation temperature. Record this solubility on your Data Sheet.

2. Recalculate the solubility of each of your diluted salt solutions, and record these solubilities along with their experimental saturation temperatures on your Data Sheet.

3. Prepare a solubility-temperature curve for your salt.

Data Sheet

ID of Unknown Salt: ________________

I. Determining the Saturation Temperature of the Original Salt Solution

Mass of weigh paper + sample (g) ________________

Mass of weigh paper (g) ________________

Mass of sample (g)* ________________

Initial volume of water added (mL) ________________

Total volume of water added (mL) ________________

Saturation temperature (^{o}C) ________________

Solubility (g salt/100 mL water) ________________

*determined as the difference of prior mass measurements.

II. Determining the Saturation Temperature of Diluted Salt Solutions

Total volume of original solution (mL): ________________

Volume of water added (mL)	Total volume of diluted solution (mL)	Saturation Temperature (^{o}C)	Solubility (g salt/100 mL H_2O)

Identification of Unknown Salt: ________________________________

Post-Lab Questions

1. How would you change the experimental procedure for this lab if the solubility of the salt was initially determined at 20^oC, followed by 35^oC, 50^oC , 65^oC , and 80^oC?

2. From the information provided in Figure 1, estimate the temperature at which 10.0 g KNO_3 dissolved in 12.5 mL of water would be a saturated solution.

3. While transferring the weighed salt to the test tube in this experiment, a student accidently spilled some of his/her sample. How will this salt loss affect the salt solubilities calculated by this student? Will they be too high, too low, or unaffected? Explain.

4. It is important to keep the test tube closed to avoid evaporation of water while heating the salt solution.
   (a) Briefly explain how a loss of water by evaporation would affect the initial calculation of the solubility of your salt.

   (b) Would this initial evaporation affect the calculated solubility of your salt at each subsequent experimental saturation temperature, or just the initial temperature? Explain.