Lab 1: Determining Solubility of an Unknown Salt at Various Temperatures

Objectives: To determine solubility of an inorganic salt in water as a function of temperature, to construct a solubility-temperature curve, and to identify the unknown salt by comparing an 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 1.2); ceramic-centered gauze heating pad; 400 mL beaker; hot plate.

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 salt water have in common? These, and many other substances that 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 natures 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 *more* 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 so that the excess salt remains undissolved. The system is dynamic; some salt particles may recrystallize as dissolved ions recombine to form a 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 dissolves. 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 will feel 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 solution increases.

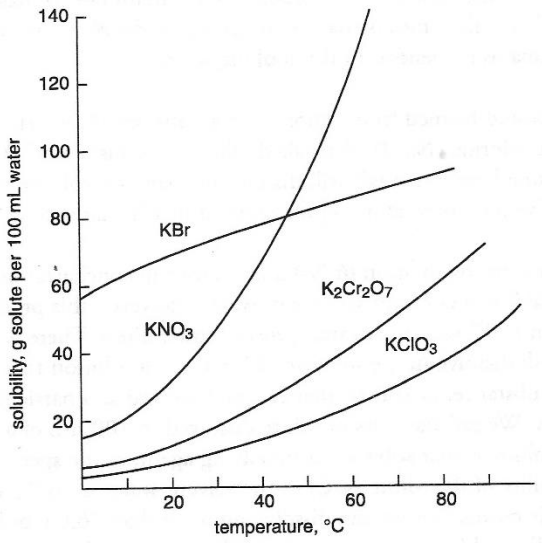


Figure 1.1. Solubility-temperature curves for various inorganic salts in water.

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 their experimental solubility-temperature curves with curves of known salts. Solubility-temperature curves of several inorganic salts are presented in Figure 1.1.

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 1.1.

$$\text{concentration of salt solution} = \left(\frac{\text{experimental mass of salt (g)}}{\text{total volume of water (mL)}} \right) \times 100 \qquad \text{(Equation 1.1)}$$

Using your calculated solution concentrations and experimentally determined saturation temperatures, you will construct a solubility-temperature curve for your salt and calculate the percent error between your experiment and the accepted value. Remember, percent error is an absolute value calculation. No matter what the final value, it is recorded as a positive number.

% error = [(accepted value – experimental value) / (accepted value)] x 100% (Equation 1.2)

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?

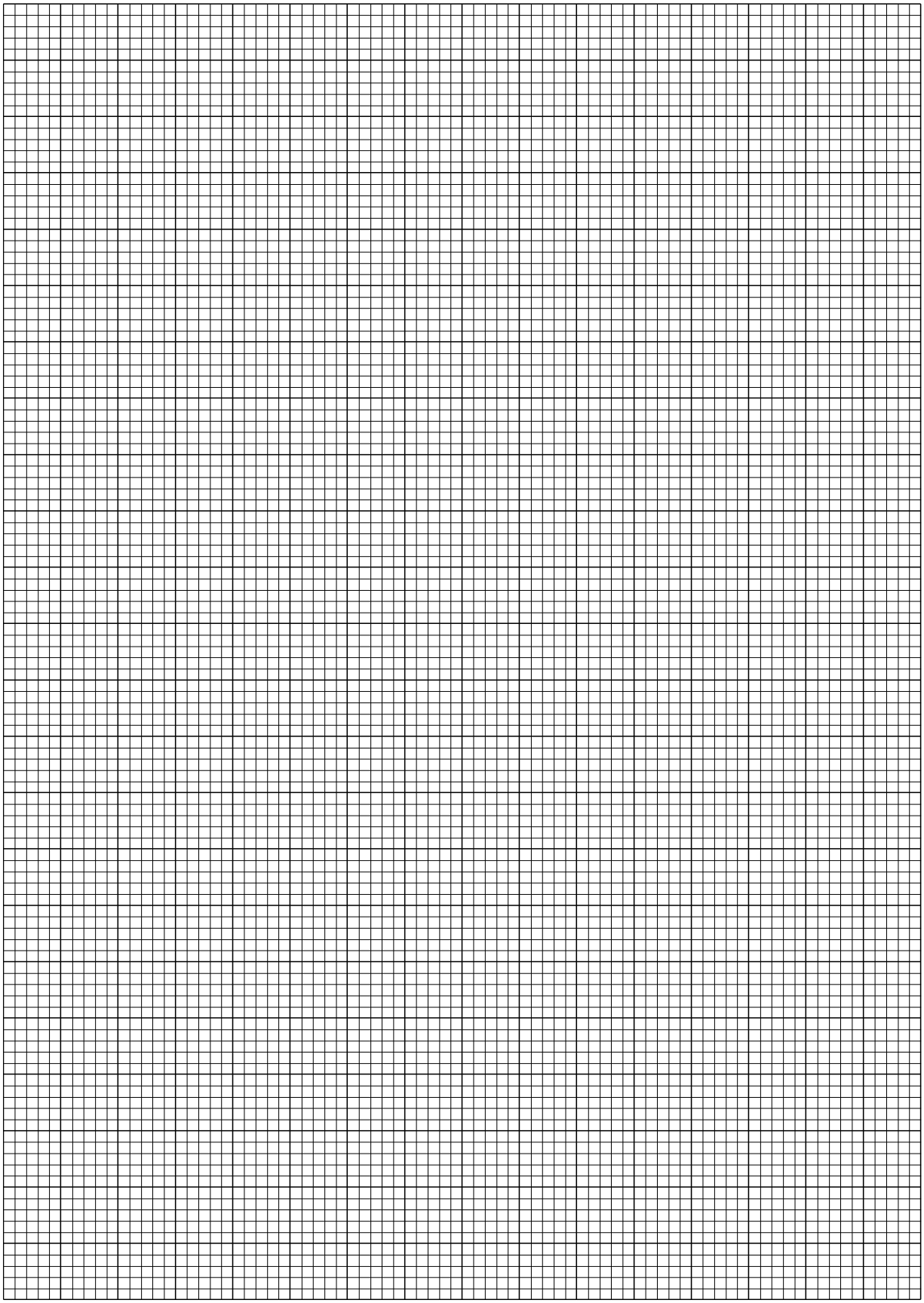
3. What factors determine the solubility of a salt in water? Explain.

4. A student collected the following temperature-solubility data for ammonium chloride (NH_4Cl) dissolved in water:

   Mass of salt sample: 2.33 g

Total Volume of Water Added	**Saturation Temperature, ^oC**	**Solubility (g salt/100 mL H_2O)**
3.00	98.0	77.7
3.58	80.0	
4.24	61.0	
4.66	51.0	
5.68	30.0	
6.13	22.0	
7.06	10.0	
7.77	1.0	

   (a) Calculate the solubility of NH_4Cl for each saturation temperature and enter the values in the table above.
   (b) Prepare a solubility-temperature curve for NH_4Cl using Excel.
   (c) From your curve, estimate the solubility of NH_4Cl at 40 ^oC.



PROCEDURE

Preparing the software

a. Log on to the computer at you station using your NIU credentials.
b. Plug in the temperature probe in channel 1 of the LabQuest interface.
c. Open "chem213: folder on the desktop.
d. Open "Chem 213 Experiment Files" folder.
e. Open "Experiment 213-1 "Determining Solubility of an Unknown Salt at Various Temperatures".

NOTE: This is an **Event Based** experiment. The software will "Keep" the data with each entry.

Experiment

1. Pick the correct size test tube and stopper based on the salt that is assigned to you.

- $KClO_3$: 20mm x 200mm tube (larger) and a number 4 one-hole stopper
- KNO_3: 16mm x 150mm test tube (smaller), and a number 2, one-hole stopper

Fill the 400 mL beaker with approximately 300 mL of deionized water, and place on the hot plate as illustrated in Figure 1.2.

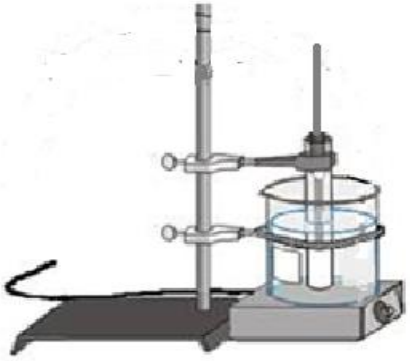


Figure 1.2. Apparatus for determining the saturation temps of salt solutions.

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

3. Turn on the hotplate. Adjust the setting to 50% of maximum setting. When the bath temperature reaches 80-85^{o}C, adjust the setting to 10-30%. Use a second thermometer to monitor the temperature water bath, it can be a glass thermometer. While the water is heating, prepare the original solution of your salt.

I. Determining the Saturation Temperature of Original Salt Solution

4. Your TA will assign you a specific salt, potassium nitrate (KNO_3) or potassium chlorate ($KClO_3$). Record the ID for your salt on your data sheet.

5. Place 5 grams of your salt, weigh 4 g if using potassium chlorate ($KClO_3$), in the weighing tray. Weigh the sample + tray and record this mass on your data sheet.

6. Carefully transfer all of your sample to your test tube. Reweigh the weighing tray and record this weigh on your data sheet.

7. Fill a clean 50 mL buret with distilled water,
If using KNO_3: dispense 3.00 mL of distilled water from the buret into the test tube containing your salt sample
If using $KClO_3$: dispense 10.00 mL of distilled water. Record the actual volume of water added to your test tube in Table 1.1 to the nearest 0.01mL.

8. Obtain a stir paddle and insert the narrow end of it through the bottom of the one-hole stopper, next to the thermometer probe. Then insert the paddle, thermometer, and the stopper into the test tube. You must be able to stir the contents of the tube using the paddle.

9. When the temperature of the water in the beaker is about 80 ºC, 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 ºC.

12. If the salt sample is not completely dissolved when the temperature of the solution has reached 80 ºC, remove the test tube from the hot water bath and add 0.50 mL of distilled water to your test tube. Record the exact total volume of water added to the test tube in Data Sheet I and in Table 1.1 under Solution 1.

13. Reheat the test tube and contents in the hot water bath to 80 ºC while stirring carefully.

14. If necessary, repeat steps 12 and 13 until your salt sample is completely dissolved in solution at 80 ºC.

15. After the salt has completely dissolved, remove the stopper and paddle from the test tube and insert the temperature probe. Click the **"Collect"** icon in the top menu bar of the Logger Pro. Use the temperature probe to continue stirring the mixture for 2-3 minutes or until the temperature reading of the probe is stable. Next, loosen the clamp holding the tube and lift and swing the test tube into a position away from the bath, Figure 1.3. Re-clamp the tube and continue stirring the contents using the probe.

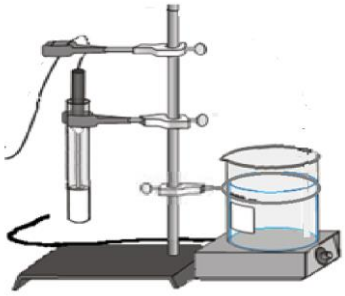
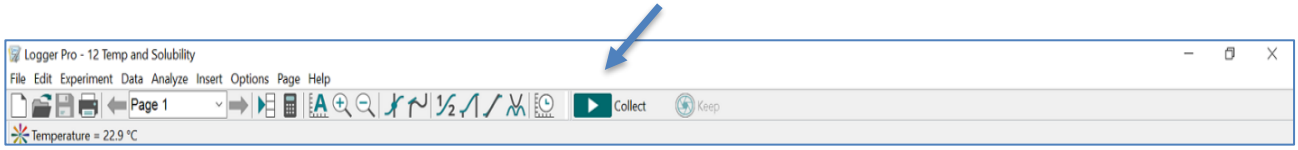
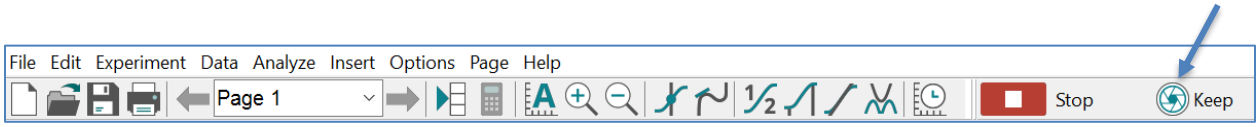


Figure 1.3 Remove test tube from hot bath

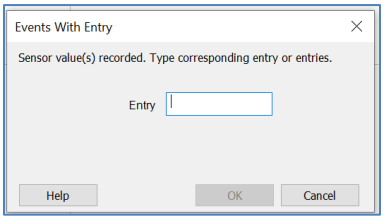


16. Continue to stir the solution in the test tube while monitoring the temperature of the solution in the test tube.

17. Carefully observe the solution. Hit the **"Keep"** icon at the temperature that salt begins to recrystallize. This is the saturation temperature. Do NOT click the "Stop" until the end of the experiment. Record this temperature in Data Table 1.1.



Enter **"1"** (for Solution 1) in the dialog box for the first crystallization temperature. You will later enter the calculated solubility in the **"Entry"** column of the data table.



II. Determining the Saturation Temperature of Diluted Salt Solutions

18. Add 0.50 mL of distilled water from the buret to the solution in the test tube, this is Solution 2. Record the volume of added water in Data Table 1.1. You will later record the **total** volume of water added to the test tube for Solution 2.

19. Place 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. Repeat step 17 to **"Keep"** the temperature of crystallization.

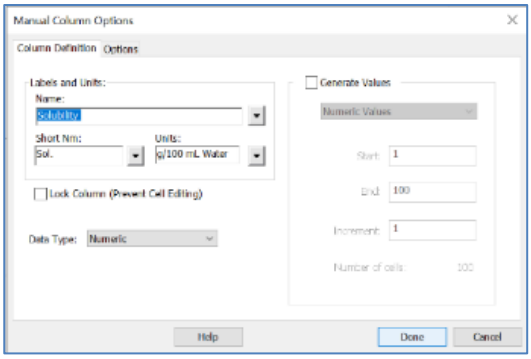
21. Enter 2, 3, … for **"Entry"** in the dialog box for the rest of crystallization temperatures.

22. Repeat steps 18 -20. If the saturation temperature does not change significantly with 0.5 mL additions of distilled water, add 1.00 mL increments.

23. If the saturation temperature falls below room temperature, replace the hot water bath with an ice bath. Add 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.

24. Press **"Stop"** icon after you have collected at least eight crystallization temperatures.
Do not exit the software until you have performed the data analysis, printed the graph, data table, and answered question 2 of post lab as instructed in the next steps.

Analyzing the Data Using Logger Pro Software



In this section, you'll input the solubility of your salt calculated at various temperatures into Logger Pro, and the software will generate a plot of the salt's solubility versus temperature.

1. Make the following changes to change the name and the units of **"Entry"** column:
   a. Right click on the data table and choose "**Column Options" > "Latest | Entry".**
   b. Enter **"Solubility"** for **"Name"**.
   c. Enter **"Sol"** for the **"Short Name"**.
   d. Enter **"g/(100 mL Water)"** for **"Units"**. Click **"Done"**.

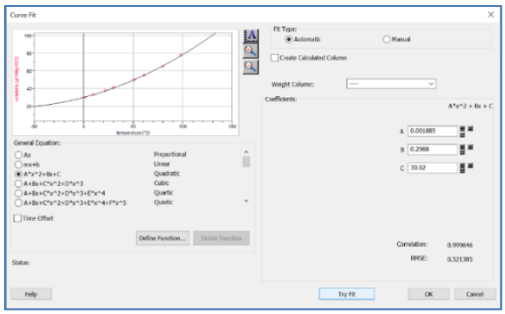
2. Calculate the solubility per 100 mL of water in Data Table 1.1 at each saturation temperature. For example, if 5.00g of salt was dissolved in total 3.00mL of water at 77.5 ºC, the solubility in 100 mL of water at 77.5 ºC is equal to:

$$Solubility\ \left(\frac{g}{100mL\ Water}\right) = \frac{5.00g\ salt}{3.00mL\ of\ water} x100 = 167\left(\frac{g}{100mL\ Water}\right)$$

3. Enter the calculated solubility at each saturation temperature in the new **"Solubility"** column of data table in Logger Pro. You can enter the value by clicking on the appropriate box. Logger Pro will generate a plot, this is the solubility plot of your salt vs. temperature.

4. You may need to use the "**Autoscale"** or manually rescale "X" and "Y" axes to see all the data points.
   To manually change the scale, double-click on the numbers on the axis and change the scale under the "Axis Options" tab.

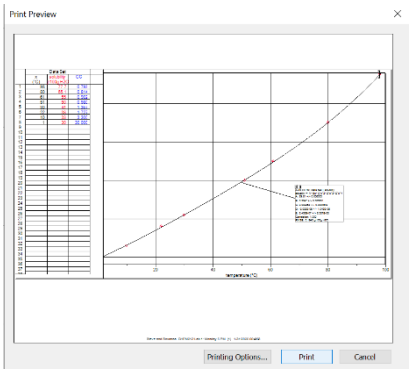
5. From **"Analyze"** menu choose **"Curve Fit"** to draw a best fit line on your graph. Try different fits to find the best fit for your graph. Hit OK when you find the best fit.



6. From **"Analyze"** menu:

a. Choose **"Interpolate"**. A vertical line will appear that can be moved on the graph for X and Y reading.

b. A box also will appear on the graph with X and Y axes readings.



7. Use the solubility plot to determine and record the solubility of your specific salt at 20 °C. This is the answer to post lab question 1A.

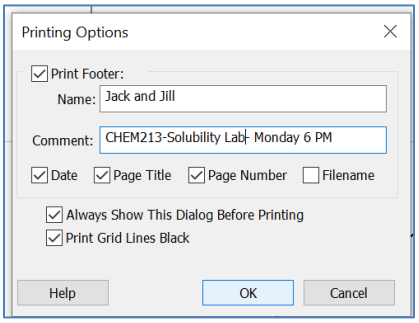
8. To print your data and graph:

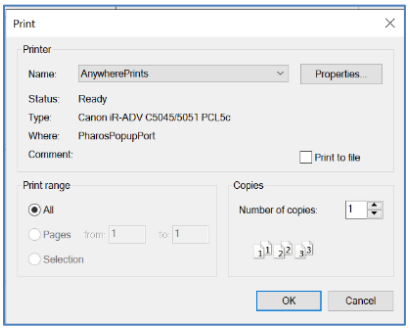
a) Go to **File > Page Set up** and choose "**Portrait"**.

b) Go to **File > Print Preview** to ensure that both the data table and the graph are in the same page. Click **"Print"**.

c) Enter your name/s and comment in the dialog box, **OK**.

d) Click the **"PrintScreen"** key on the keyboard, save the image and email it to each person in the group. Every group member can later print the image.





9. Attach or upload a copy of the data/graph to your lab report.

10. Exit the Logger Pro software. Do not save the changes.

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

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

CALCULATIONS

1. Calculate the solubility of your salt (in grams of salt per 100 mL of water) at the original saturation temperature. Record this solubility in the **"Solubility"** column in Loger Pro.

For example, if 5.00g of salt was dissolved in total 3.00mL of water at 77.5 ºC, the solubility in 100 mL of water at 77.5 ºC is equal to:

$$Solubility\ \left(\frac{g}{100mL\ Water}\right) = \frac{5.00g\ salt}{3.00mL\ of\ water} x100 = \ 167\left(\frac{g}{100mL\ Water}\right)$$

2. Calculate the solubility of each of your diluted salt solutions and record these solubilities in the **"Solubility"** column in Loger Pro along with their experimental saturation temperatures.

3. Print or screenshot a copy of solubility-temperature graph from Logger Pro.

Data Sheet

ID of 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 (^oC) _____

Solubility (g salt/100 mL water) _____

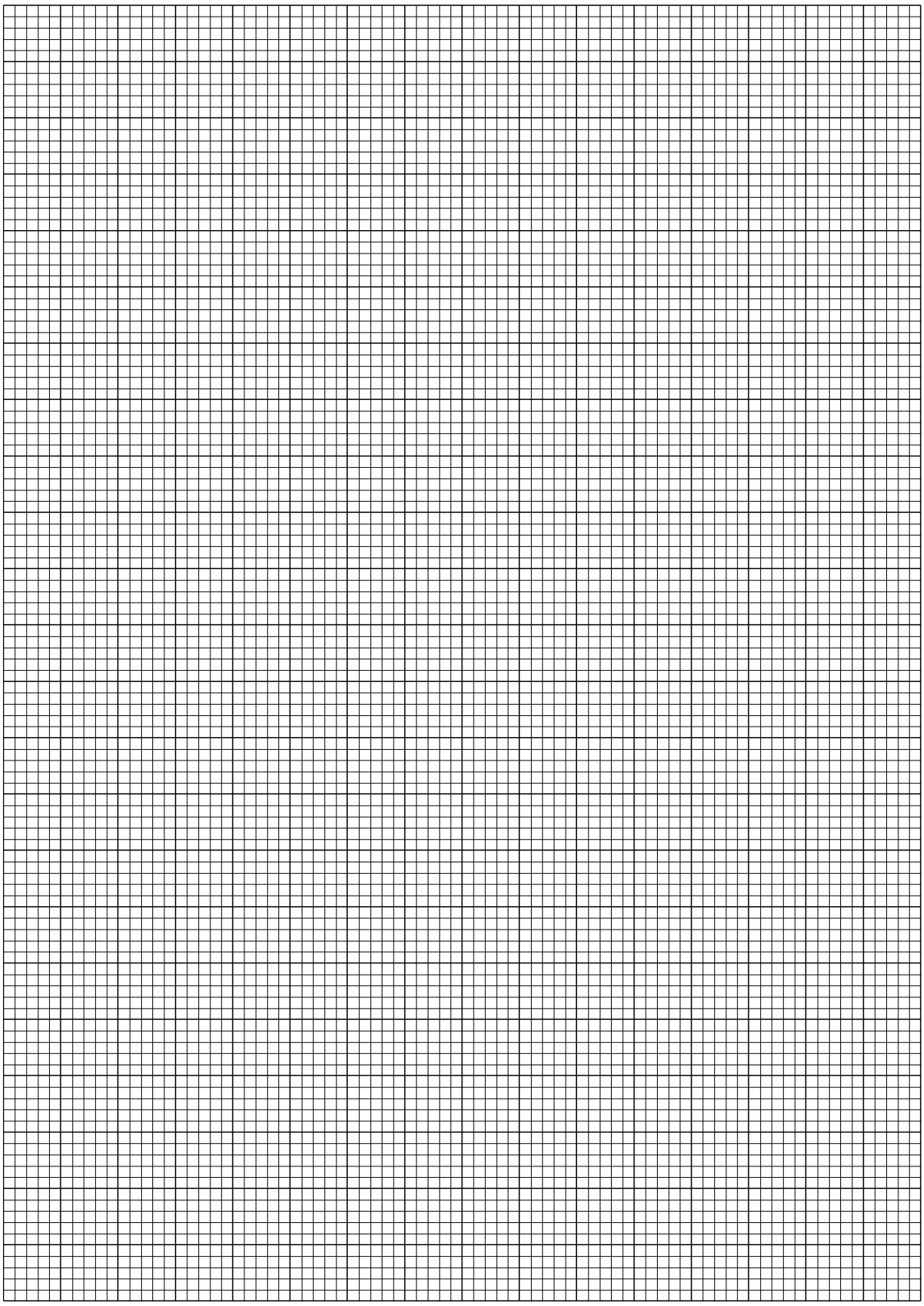
*determined as the difference of prior mass measurements

II. Determining the Saturation Temperature of Diluted Salt Solutions

Total volume of original solution (mL): _____

Data Table 1.1 Solubility of Diluted Salt Solutions

Solution #	**Volume of Water Added (mL)**	**Total Volume of Diluted Solution (mL)**	**Saturation Temperature (^oC)**	**Solubility (g salt/100 mL H_2O)**
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				



Post-Lab Questions

1. A. Determine the solubility for your specific salt at 20°C.
   B. Determine the % error for your specific salt at 20°C.
   (Accepted values at 20°C: KNO_3 – 33 g/100 mL; $KClO_3$ – 7.3 g/100 mL)

2. From the information provided in Figure 1.1, 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 and unknowingly spilled some of their 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) If a loss of water occurs unknowingly, briefly explain how this 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.