



Laboratory **1**

(SOL)

Variation of Solubility with Temperature and Solvent

Expected Learning Outcomes

- Explain the difference between a molecule's solubility in solvents of varying polarities by describing solute-solvent interactions
- Investigate the effect temperature has on solubility
- Interpret and analyze quantitative solubility data expressed in multiple units (mole fraction and molality)
- Use Microsoft Excel to manipulate data, perform calculations within the spreadsheet format, and produce informative graphs

Introduction

Solubility is an important topic in chemistry and fundamental to many life processes. A solid understanding of solubility is necessary for scientists, health professionals, and engineers alike. Solubility is a physical property that can be quantified. Through daily experiences we know that some substances can become more or less soluble if we change the surrounding conditions. For example, if you like to sweeten your tea, you know that sugar will dissolve much more easily in hot tea than it will in iced tea. In this lab, we will consider factors that affect solubility.

This is a two-week lab. On Day 1, you will collect data and follow the procedure outlined for the experiment. Before Day 2, you will complete the online pre-lab for this experiment. Upon submission, the pre-lab will provide you with a set of data; copy it into your notebook. On Day 2, you will create a graph including both the data you collected in lab on Day 1 and the data provided in the pre-lab. Bring a laptop or tablet that can run Microsoft Excel.

Discussion

In previous chemistry courses, our discussion of solubility was mostly *qualitative* and focused on the use of solubility tables. These tables summarize the rules for solubility and allow us to broadly classify compounds as “soluble” or “insoluble.” In this course, we will expand our understanding of solubility to be *quantitative*. While two compounds may be soluble in the same solvent, one compound likely is *more* soluble than the other.

We will learn that different factors can influence how soluble something is. Factors that affect the solubility include:

1. temperature effects
2. solute–solvent interactions
3. in gaseous systems, pressure effects

In this lab, you will be looking at the influence of temperature effects (Part A) and solute–solvent interactions (Part B) on the solubility of a compound.

Temperature Effects

It is no surprise that solubility depends on temperature. The relationship between temperature and solubility for several substances is shown graphically in Figure 1.1. As illustrated, solubility of an ionic solute in water generally increases with increasing temperature (although there are exceptions). Note two other characteristics about the data displayed. First, each substance has a unique curve, and secondly, dependence of solubility on temperature is generally not a linear relationship. Solubility is an experimentally determined property.

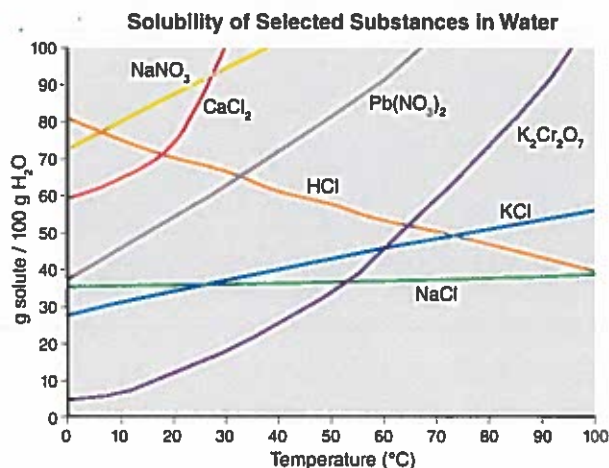


Figure 1.1 The solubility of different substances in water as a function of temperature. The solid lines are conditions for a saturated solution. The region above the line represents a saturated solution, and the region below the line an unsaturated solution for a particular temperature.

Explanation of the effect of temperature on solubility is in terms of competing processes and energies. Consider formation of an aqueous solution of potassium iodide:



Before the solution can form, the solid KI must be pulled apart—forces of attraction between K^+ ions and I^- ions must be overcome. This process (separation) requires energy. The separated ions are then surrounded by layers of water molecules—they become hydrated. This process (hydration) evolves energy because of the electrostatic attractions between the charged ions and the polar water molecules.

Spot Check 1.1: Is the separation process endothermic or exothermic? What about the hydration process? What is the sign of ΔH for each?¹

A much smaller amount of energy is involved in separating water molecules from each other, and it is included in the hydration energy. The process of solution can be summarized using an energy diagram, as shown in Figure 1.2.

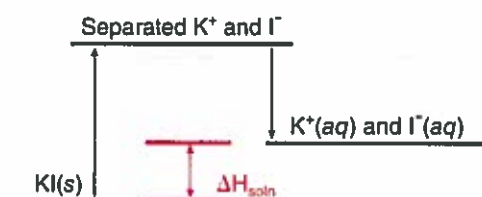
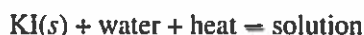


Figure 1.2 Energy level diagram for the process of a potassium iodide solution.

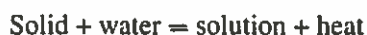
The overall energy change for the process of solution, ΔH_{soln} , is the difference between the energy of the solution [$K^+(aq)$ and $I^-(aq)$] and the energy of the solute [$KI(s)$] to be dissolved. In this case, ΔH_{soln} is positive and energy is required when KI dissolves in water, an endothermic process. This can be indicated by writing heat as a reactant in the equation for the solution process:



If we increase the temperature by adding heat, we are “supplying a reactant” so more solution should form, provided both KI and water are available.

Spot Check 1.2: Does the solubility of KI increase or decrease with increasing temperature?

The strength of attraction between the ions in the solid and the strength of hydration both depend on the size and charge of the ions. If the energy of hydration is greater than the energy required to pull the solid apart, the overall heat of the solution will be negative ($\Delta H_{\text{soln}} < 0$) and heat will be evolved when the solution forms.



¹ Spot Check questions are not graded. These are here to help guide you through the lab—they can be discussed with your group and lab instructor.

In this case, increasing the temperature “supplies a product,” which is a “reactant” for the reverse reaction. Increasing the temperature causes the reverse reaction; solid crystallizes out of solution and solubility decreases. These ideas are explored further in Chapters 15 and 17 of your textbook.

Solute–Solvent Interactions

Solubility also depends on the nature of the solute and the solvent. The fact that each substance in Figure 1.1 has a unique curve emphasizes this phenomenon. Similarly, if you use the same solute and change the solvent, you would also expect unique behavior. A generalization can be made about solubility, solutes, and solvents: substances with similar intermolecular forces of attraction tend to be soluble in each other. This is stated concisely as “like dissolves like.”

For a solution to form, intermolecular attractions between particles in the *solute* must be overcome, attractions of *solvent* particles for each other must be overcome, and new attractions must exist *between solute and solvent*. In other words, solute–solvent attractions must be strong enough to compete with solute–solute and solvent–solvent attractions. Chapter 11 of your textbook includes a discussion of the various intermolecular forces that exist in solutions.

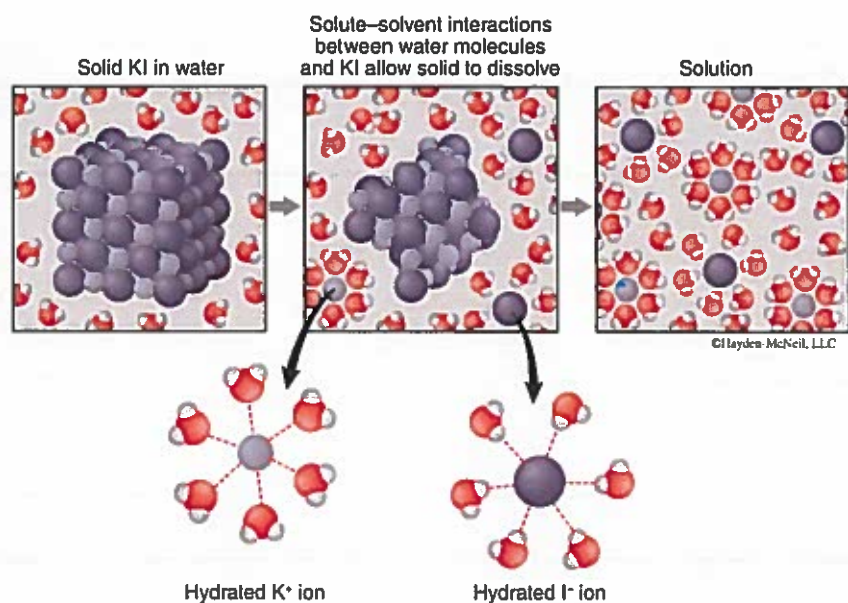


Figure 1.3 The dissolution process of KI in water. The intermolecular forces of attraction between water molecules and attractions between the ions in KI must be overcome to create a solution. This is achieved through the stronger interaction between K^+ and I^- ions with water.

Consider again the process of dissolving potassium iodide in water, as shown in Figure 1.3. Attractions between particles in the solute are electrostatic attractions between positive ions and negative ions, K^+ and I^- . Attractions between solvent particles (H_2O molecules) are hydrogen bonding, dipole–dipole, and London dispersion forces. The ion–dipole forces of attraction between K^+ ions and the partial negative region of a water molecule, and

between I^- and the partial positive region of a water molecule are strong enough to compete with the solute-solute and solvent-solvent attractions. The solution forms. The solubility of KI is 128 g per 100 g H_2O at 0 °C.

If we try to dissolve KI in a nonpolar solvent such as carbon tetrachloride, CCl_4 , the weak London forces of attraction between ions and CCl_4 molecules cannot compete with the ion-ion forces in KI. Potassium iodide is essentially insoluble in CCl_4 .

Now consider dissolving I_2 in water and also in CCl_4 . London forces hold the solid I_2 together. The only attraction between the solute and solvent is the London force. Water has very weak London forces; carbon tetrachloride has relatively strong London forces. The solubility of I_2 is 100 times greater in CCl_4 than in water. Again, "like dissolves like."

Overview

A saturated solution is a solution in which both dissolved and undissolved solute are "in equilibrium" with each other. That is to say, a saturated solution contains as much solute as it can hold while in equilibrium with excess undissolved solid solute. We will make use of saturation—the point at which no further solute can dissolve in solution—to quantify the solubility of our compound. Solute will be dissolved in heated solutions and then allowed to cool. When the temperature is such that solute begins to crystallize, the saturation equilibrium is established. When taking measurements you will be finding the temperature at which the first crystal forms. Known amounts of solute and solvent are heated and stirred until all of the solute dissolves. The solution is stirred while it cools, and watched closely until the first crystals form. The temperature is recorded. Additional solvent is added to the same test tube, and the heating and cooling processes are repeated. The temperature is recorded at each concentration. This is the general procedure for each part of the experiment.

You will use the same solute for all parts of the experiment: oxalic acid dihydrate, $H_2C_2O_4 \cdot 2 H_2O$, a solid white organic acid. For Part A, the solvent is water. The solvent for Part B is a 70:30 water/dioxane solution. As it does not contain a molecular dipole, dioxane is a nonpolar molecule (see Figure 1.4). It is soluble in water because the oxygen atoms in the molecule have unshared pairs of electrons available to form hydrogen bonds with water molecules. While hydrogen bonds typically form between substances with an H atom bonded to N, O, or F atoms, these high-strength interactions can form when a substance with strong partial charges is present. The partial negative charge of the O atoms in the dioxane can hydrogen bond with the electron-deprived H atoms in molecules of H_2O .

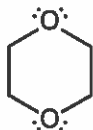


Figure 1.4 Structure of dioxane ($C_4H_8O_2$).

Data Analysis

When studying solubility, it helps to quantify how much solute is able to dissolve in a given solvent. Solubility is the amount of substance that will dissolve in a given amount of solvent or solution. Common units of solubility are g solute per 100 g solvent. Another concentration unit that we will use for solubility in this experiment is mole fraction, X .

$$X_{\text{solute}} = \frac{\text{mole solute}}{\text{mole solute} + \text{mole solvent}}$$

For each solution, determine grams of solute in 100 g solvent using dimensional analysis. For aqueous solutions, assume a density of water of 0.9975 g/mL at 23 °C. At the same temperature, density of the water/dioxane solvent is 1.023 g/mL.

Example A: If 7.0510 g of solute is dissolved in 20.00 mL of the water/dioxane solvent, what is the solubility in units of g solute per 100 g solvent?

$$20.00 \text{ mL solvent} \cdot \frac{1.023 \text{ g}}{1 \text{ mL}} = 20.46 \text{ g solvent}$$

$$\frac{7.0510 \text{ g solute}}{20.46 \text{ g solvent}} = \frac{x \text{ g solute}}{100 \text{ g solvent}}$$

Then rearrange to solve for x :

$$100 \text{ g solvent} \cdot \frac{7.0510 \text{ g solute}}{20.46 \text{ g solvent}} = 34.46 \text{ g solute per 100 g solvent}$$

After completing the pre-lab for SOL (before Day 2), you will be given data similar to what you would obtain in lab: the mass of solute used, total volume added, and the temperature at which crystals form. This data will be for potassium dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$, an orange inorganic salt. This data will be included on your graph, and you will compare it to the data you collected for oxalic acid. After calculating the concentration and solubility, you will begin the construction of a graph with the help of your lab instructor.

The graph, constructed using Excel, will have solubility on the y-axis and temperature on the x-axis. Data for aqueous solutions of oxalic acid (your data taken in Part A) should be plotted as one set of data and fall along a single curve. Data for the oxalic acid in the mixed solvent (your data taken in Part B) should form another curve. Data for aqueous solutions of potassium dichromate (provided after completing and submitting the pre-lab) should form a third curve. Plot all sets of data on the same graph, add trendlines (they will not be linear; an exponential or polynomial fit generally works well), and display the equation for the line and R^2 value.

Next, calculate the mole fraction of the solute for the data for both compounds in aqueous solution. Construct a second graph of temperature on the y-axis versus mole fraction on the x-axis. Data for aqueous solutions of oxalic acid (your data taken in Part A) should be plotted as one set of data and fall along a single curve. Data for aqueous solutions of potassium dichromate (provided after completing and submitting the pre-lab) should form a second curve. Plot both sets of data on the same graph, add a trendline (a logarithmic, \ln , fit works well) to each data set, and display the equation for the line and R^2 value.

Example B: If 5.4942 g of $\text{K}_2\text{Cr}_2\text{O}_7$ dissolves in 20.00 mL of water, what is the mole fraction?

$$X_{\text{solute}} = \frac{\text{mole solute}}{\text{mol solute} + \text{mol solvent}}$$

$$\text{moles solute} = 5.4942 \text{ g K}_2\text{Cr}_2\text{O}_7 \cdot \frac{1 \text{ mol}}{294.19 \text{ g}} = 0.018676 \text{ moles}$$

$$\text{moles solvent} = 19.95 \text{ g H}_2\text{O} \cdot \frac{1 \text{ mol}}{18.02 \text{ g}} = 1.107 \text{ moles}$$

$$X_{\text{solute}} = \frac{0.018676 \text{ mol}}{0.018676 \text{ mol} + 1.107 \text{ mol}} = 0.01659$$

Materials Required

Equipment

5-mL pipet
10-mL pipet
hot water bath (600-mL beaker)
cold water bath (400-mL beaker)
Bunsen burner
thin-walled rubber tubing
ring stand, ring
wire mesh
solubility apparatus

Chemicals

oxalic acid dihydrate, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2 \text{H}_2\text{O}$
(13 g/student)
70:30 water:dioxane solution, $\text{H}_2\text{O}:\text{C}_4\text{H}_8\text{O}_2$
(50 mL/student)

Common Equipment

analytical balance

Cautions

Dioxane is a suspected carcinogen. The vapor might be irritating to lungs and mucous membranes. The student hoods must be on. Gloves are highly recommended. Oxalic acid is poisonous. If these substances come into contact with your skin or clothing, flood the affected area with water. Stir samples gently, as vigorous stirring will result in the test tube breaking and a loss of sample. Be careful to avoid burns from the ring, beaker, and open flame. Never leave an open flame unattended. Goggles must be worn at all times.

Procedure

DAY 1

Part A. Determine solubility in water as a function of temperature.

1. Obtain the solubility apparatus from your lab instructor. It consists of a thermometer in a rubber stopper in a large test tube. The coiled end of the wire stirrer should encircle the thermometer, as shown in Figure 1.5.



Figure 1.5 Solubility apparatus.

2. Clean and dry the inside of the test tube. Add between 6.0 and 6.2 g of oxalic acid, weighed on the analytical balance, and add it to the test tube. Use a pipet to deliver 10.00 mL of distilled water to the test tube, rinsing down any solid stuck to the side of the tube.
3. Set up a hot water bath. Connect a Bunsen burner to a gas line and place it in your hood. About 1 inch from the top of your burner, attach a metal ring to your ring stand and place wire mesh over the ring. Fill a 600-mL beaker about $\frac{3}{4}$ full of tap water and place it on the mesh. Light the burner (see Appendix B for more information) and heat the water until it boils. Never leave the flame unattended; turn the burner off if you need to walk away.
4. Dissolve the sample completely by heating the test tube in the hot water bath. Periodically remove the test tube from the beaker (using the towel in your locker to avoid burns) and gently swirl it. Stir the solution with the wire stirrer frequently to speed up the process; do NOT use the thermometer to stir. The wire stirrer should move up and down freely without resistance. When you stir the solution with the wire stirrer, make sure the coil encircles the thermometer and use slow up-and-down movements. Rapid stirring can cause the thermometer or the test tube to break.
5. After the solid is completely dissolved, remove the test tube from the water bath and allow the solution to cool, while gently stirring, to maintain a uniform temperature and concentration. Record the temperature at which crystals are first observed.

6. Use a pipet to deliver an additional 5.00 mL of distilled water to the test tube. Repeat Steps 4 and 5.
7. Repeat Step 6 two more times for a total of four determinations with different volumes. Ice should **not** be used in the first few trials. Be careful to stir all of the solution, especially when a large volume is used. If colder temperatures are needed for later trials, place your test tube in an ice/water bath *after it has cooled to room temperature*. Ice is located in the hallway across from the storeroom window.

Spot Check 1.3: If you leave the lab room to get ice, what equipment will you need and what must stay behind? Can chemicals leave the lab? Think back to the safety video's section on glove etiquette; can you touch the door knob with a gloved hand? What does this imply about wearing gloves in the hallway?

8. After all four determinations, discard the solution in your personal waste beaker.

Part B. Determine solubility in 70:30 water:dioxane as a solvent.

9. Clean and dry the inside of the test tube. Weigh between 6.0 and 6.2 g of oxalic acid on the analytical balance and add it to the test tube.
10. Use a pipet to deliver 10.00 mL of 70:30 water:dioxane solution to the test tube. Note that the volume of solvent used in Part B differs from the volume used in Part A.
11. Dissolve the sample completely by heating the test tube in the hot water bath. Periodically remove the test tube from the beaker and gently swirl it. Stir the solution with the wire stirrer frequently to speed up the process; do NOT use the thermometer to stir. When you stir the solution with the wire stirrer, make sure the coil encircles the thermometer and use slow up-and-down movements. Rapid stirring can cause the thermometer or the test tube to break.
12. After the solid is completely dissolved, allow the solution to cool, while gently stirring, to maintain a uniform temperature and concentration. Record the temperature at which crystals are first observed. If colder temperatures are needed for later trials, place your test tube in an ice/water bath *after it has cooled to room temperature*.
13. Use a pipet to deliver an additional 10.00 mL of 70:30 water:dioxane solvent to the test tube. Repeat Steps 11 and 12. Again, please note that the volume of solvent used in Part B is different than in Part A.
14. Repeat Step 13 two more times for a total of four determinations. Discard the solution in your personal waste beaker and clean and dry the test tube.

Waste Disposal

Collect the waste in a beaker at your desk. All solutions containing oxalic acid must be disposed of in the organic solvents beaker. Fill in the Chemical Waste Disposal Form, and alert your lab instructor if the beaker is more than half full and ready for disposal. Your lab instructor will dispose of the total volume in the appropriate container.

DAY 2

As an OSU student, you have the ability to download a free copy of Microsoft Office (this includes Word, Excel, and many other programs).

For help, visit: office365.osu.edu
Additional help may be found at the bottom of the webpage.

Complete the following tasks using the data you collected on Day 1 and the data provided after completing and submitting the pre-lab. A summary of the graphs you need to create is in the Data Analysis section (page 10), but a more specific procedure is provided here. You may consult your peers as you work through the instructions; if you collectively have questions, ask your lab instructor for additional guidance. **All calculations must be done in Excel**, so be sure to download this program before arriving for Day 2.

1. Input the data you collected (Total Volume and Temperature) in lab on Day 1 for Part A into a new spreadsheet in Excel, placing each in its own column. Do not worry about formatting the column headers.
2. In a new column, calculate the mass of water present in each sample using the density given in the lab manual (page 10) and the volume you input to your spreadsheet. Excel will perform these calculations for you if you type in the correct function. For example, B2 is one cell that contains a volume. In cell C2, you could enter the following formula:

$$= B2*(\text{density of solvent})$$

$$= B2*(0.9975)$$

Note that units are not used in the formula command.

3. Excel does not automatically report the correct number of significant figures. Adjust the number of significant figures shown by changing the number of decimal places displayed; the cell format should be changed from "General" to "Number" to allow this. Significant figure rules are provided in the lab manual starting on page D-2.
4. In another column, determine the solubility in units of grams of solute per 100 grams of solvent. An example of this calculation is provided in the Data Analysis section. Be sure to report the correct number of significant figures. Follow the example given in Step 2 to allow Excel to perform this calculation for you.
5. Construct a graph with solubility on the y-axis and temperature on the x-axis. The "Insert" tab will allow you to add a chart. First, select the x and y data you wish to plot by highlighting it in the spreadsheet. Next, select the "Scatter" plot option to create your chart. A small plot—your "solubility" graph—will appear.
6. Two other series of data will need to be added to your solubility graph. Add the mixed solvent data you collected in Part B of Day 1 as a "new series." Adding a "new series" can be done in many ways, but is often done through "Select Data." Finally, add the data for potassium dichromate in water provided in the pre-lab as a third series. You should now have one solubility graph with three sets of data on it: oxalic acid in water, oxalic acid in the 70:30 water:dioxane, and potassium dichromate in water.

7. Move the solubility graph to a new sheet; to do so, select "Move Chart" and the "New Sheet" option to create Chart1. Your solubility graph will now be a full page. Format your graph appropriately and add trendlines for all series and display R^2 values; refer to the Guide for Success in the General Chemistry Laboratory and Appendix D for graphing requirements.
8. Return to your original data spreadsheet. Using a new column, calculate the mole fraction of the solute for all "water as a solvent" samples. You will have two series of data this time: oxalic acid in water (your data from Part A) and potassium dichromate in water (provided in the pre-lab). An example of the calculation to determine mole fraction is provided in the Data Analysis section. Be sure to report the correct number of significant figures.
9. Construct a second graph with temperature on the y-axis versus mole fraction on the x-axis, making sure to add both series. Move the graph to a new sheet; add a trendline and display R^2 values for both series, and format the graph according to the graphing guidelines.
10. Both of your graphs will now need to be added to your Report Template for submission. Both graphs display data most accurately in the "landscape" orientation; this allows the data to be "spread out." Two pages of the Report Template will be turned to "landscape" orientation; add your graphs to these pages. Check that all formatting remains in your final graphs.
11. Have your lab instructor check your graphs before leaving for the day. Incorporate their feedback, if needed.

Points to Consider

- Based on your graph, how does the solubility of oxalic acid trend as a function of temperature? Of potassium dichromate?
- What does your mole fraction graph show, with respect to the “water as a solvent” data for oxalic acid? Explain why the data does not fit a linear trend.
- Were the observed solubility trends as expected based on the material you have read about in the text and learned in lecture? Explain.
- Which solvent (water or water:dioxane) *should* oxalic acid have the higher solubility in? Why? What solvent *did* oxalic acid have a higher solubility in? Do your results match your prediction?
- Compare oxalic acid and potassium dichromate. Describe the attractive forces present in both solutes and solvents (water and 7:30 water:dioxane). Based on these, predict if potassium dichromate would be more or less soluble in the mixed solvent.
- In this experiment, everyone individually examined the solubility of oxalic acid by visually determining when crystals form in solution. Do you think if you and your neighbor had the same mass of solute and same volume of solvent that you would record the same temperature at which crystals form? What type of error would result if the data from multiple scientists was combined?

Use these Points to Consider to shape the Discussion section of your report. For full credit, address each point thoroughly and support your conclusions with data. The Discussion section should be presented as a few cohesive paragraphs. No subheadings are necessary, and should not be included.

What is the Data to Collect section?

All data collected in lab is written in your notebook. The Data to Collect section outlines the data, calculations, and observations you should be recording in your lab notebook. The section is labeled and located in your lab manual at the end of most experimental procedures. It does not necessarily match what values you will need to include in your Report Quiz or lab report—the quiz itself or your Report Template will include the precise data you need to report. All data collected must:

- Include the correct number of significant figures and units.
- Be recorded directly into your notebook—NOT onto the Data to Collect pages or on a computer.
- Include work for calculated values—also done directly in your notebook.

Data to Collect

Variation of Solubility with Temperature and Solvent

OXALIC ACID DATA

Part A. Determine solubility in water as a function of temperature.

Mass of solute, g _____

Addition	Total Volume (mL)	Temp at Which Crystals Form (°C)	Concentration (g Solute/100 g Solvent)	Mole Fraction
1				
2				
3				
4				

Part B. Determine solubility in 70:30 water:dioxane as a solvent.

Mass of solute, g _____

Addition	Total Volume (mL)	Temp at Which Crystals Form (°C)	Concentration (g Solute/100 g Solvent)
1			
2			
3			
4			

POTASSIUM DICHROMATE DATA

Data for water as a solvent (obtained before arriving for Day 2 after completing and submitting the pre-lab)

Mass of solute, g _____

Total Volume (mL)	Temp at Which Crystals Form (°C)	Concentration (g Solute/100 g Solvent)	Mole Fraction