Experiment 21

Solubility

Pre-Lab Assignment

Before coming to lab:

- Read the lab thoroughly.
- Answer the pre-lab questions that appear at the end of this lab exercise.

Purpose

A series of standardized dilutions of copper(II) sulfate pentahydrate ($CuSO_4 \cdot 5\ H_2O$) will be used to prepare a Beer's Law plot with spectrometry to find the relationship between concentration and absorbance. The average volume of 10 drops dispensed by a plastic pipette will be calibrated. With this, a series of solutions of $CuSO_4 \cdot 5\ H_2O(aq)$ will be prepared at different temperatures to determine whether the relationship between solubility and temperature is inverse or direct.

Background

Solubility is the measure of how much of a substance will dissolve in asset amount of solvent. Compounds that remain as solids in aqueous solution are classified as insoluble, whereas compounds that dissolve into separate ions in aqueous solution are classified as soluble. The degree to which a compound dissolves in aqueous solution is known as its solubility and is dependent on a number of factors, including temperature, concentration, and pH.

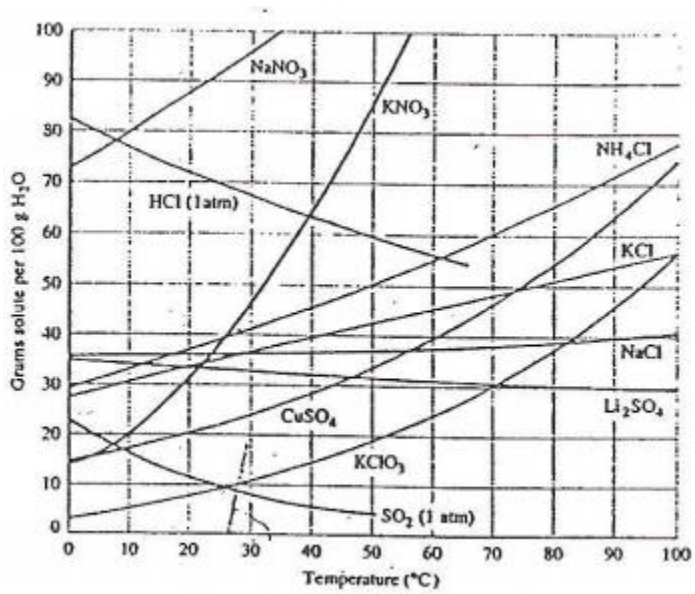


Fig. 1: Solubility of solids and gases at different temperatures

In general, the solubility of solids increases with increasing temperature whereas the solubility of gases decreases with increasing temperature (Fig. 1). The solubility of a compound can be measured in terms of its concentration, and its concentration can be measured via spectrometry. In a spectrometer, a beam of light is shined onto the solution which absorbs some of its intensity while the rest of the light passes through. The instrument measures the ratio of light allowed to pass through the sample against the its original intensity, giving readings in units of percent transmittance (%T). Since percent transmittance is based on an arbitrary scale of 0-100%, it is usually converted into absorbance (A) by Eqn. 2. Absorbance is a decimal and a unitless value.

$$A = -\log(\%T/100) \qquad \text{Eqn. 2}$$

Beer's Law states that the absorbance of a solution is related to its concentration by Eqn. 3.

$$A = \varepsilon \, l \, C \qquad \text{Eqn. 3}$$

Here, ε is the molar absorptivity constant (in M^{-1} cm^{-1}) which is individual to the identity of the solution, l is the path length of the light source inside the spectrometer (usually the width of the sample container, in cm) and C is the concentration of the solution (in M). Since ε and l are constant so long as the same compound and same spectrometer are used, a plot of concentration versus absorbance should be linear, giving a mathematical equation that can be used to determine the concentration of an unknown solution from its absorbance.

Example Problem: Determining the Concentration of an Unknown Solution

A previous set of standardized solutions of $CuSO_4 \cdot 5\ H_2O(aq)$ gave a trendline for concentration (x-axis) versus absorbance (y-axis) of $y = 0.8895x + 0.0717$. A new solution was made by adding 10 drops of a saturated $CuSO_4 \cdot 5\ H_2O(aq)$ from a calibrated plastic pipette to 2.50 mL of deionized water. Its absorbance was measured to be 0.354. The pipette was calibrated to dispense an average of 0.534 mL per 10 drops. Calculate the concentration of the original 10 drops of saturated solution.

Step 1: Convert absorbance to concentration of diluted solution

$$0.354 = 0.8895x - 0.0717$$
$$0.426 = 0.08895x$$
$$x = 0.479\ M$$

Step 2: Use $M_1V_1 = M_2V_2$ to find the original concentration

$$(0.479\ M)(2.50\ mL + 0.534\ mL) = (M_2)(0.534\ mL)$$

$$M_2 = 2.72\ M$$

Procedure

Part I: Calibration of the Plastic Pipette

1. Weigh the smallest size clean, dry Erlenmeyer flask with stopper. Record the mass in your data sheet.

2. Fill a small beaker approximately half-full with deionized water.

3. Fill your plastic pipette approximately a quarter full with the deionized water from the beaker in Step 2. Hold the dropper vertically and practice dispensing and counting single drops of liquid from it.

4. Once confident, dispense exactly 10 drops of deionized water into the empty Erlenmeyer flask from Step 1 and stopper it. Record the mass in your data sheet.

5. Add exactly 10 more drops of deionized water to the Erlenmeyer flask and stopper it. Record the new mass in your data sheet.

6. Add exactly 10 more drops of deionized water to the Erlenmeyer flask and stopper it. Record this third mass in your data sheet.

7. Record the temperature of the sample of deionized water used with a glass thermometer.

8. Squirt the pipette dry for later use.

9. Look up the accurate density of water at the temperature you recorded in Step 7.

10. Find the masses of each individual 10-drop portion of deionized water. Use the density you researched in Step 9 to convert each 10-drop mass to volume. Find the average volume of 10 drops dispensed from your plastic pipette.

Part II: Determination of Standard Solutions

1. Obtain six clean, dry small test tubes. Each one should be without scratches, stains, or other blemishes. Number the test tubes 1-6 as close to the top as possible.

2. Fill each of the test tubes with the following:

#1: approximately 3 mL of deionized water. This will serve as your "blank".
#2: approximately 3 mL of 0.200 M $CuSO_4 \cdot 5\ H_2O(aq)$

Note: do not use a graduated cylinder for the following measurements. Use a volumetric pipette. The pipette has higher precision, which is needed for this part of the experiment.

#3: 4.00 mL of 0.200 M $CuSO_4 \cdot 5\ H_2O(aq)$ + 1.00 mL deionized water
#4: 3.00 mL of 0.200 M $CuSO_4 \cdot 5\ H_2O(aq)$ + 2.00 mL deionized water
#5: 2.00 mL of 0.200 M $CuSO_4 \cdot 5\ H_2O(aq)$ + 3.00 mL deionized water
#6: 1.00 mL of 0.200 M $CuSO_4 \cdot 5\ H_2O(aq)$ + 4.00 mL deionized water

3. Calculate the concentration in Molarity of Tubes #3-6.

4. Obtain a SpectroVis Spectrophotometer kit and a LabQuest 2 Interface or lab computer. The kit will include a spectrophotometer and two small plastic cuvettes with caps. Do not lose these.

5. If using a lab computer, ensure that LoggerPro is running. Plug in the spectrophotometer via the USB port. The interface or computer should automatically detect the sensor.

6. To calibrate the spectrophotometer, .go to the Experiment menu and select Calibrate, then Spectrometer (Fig. 2).

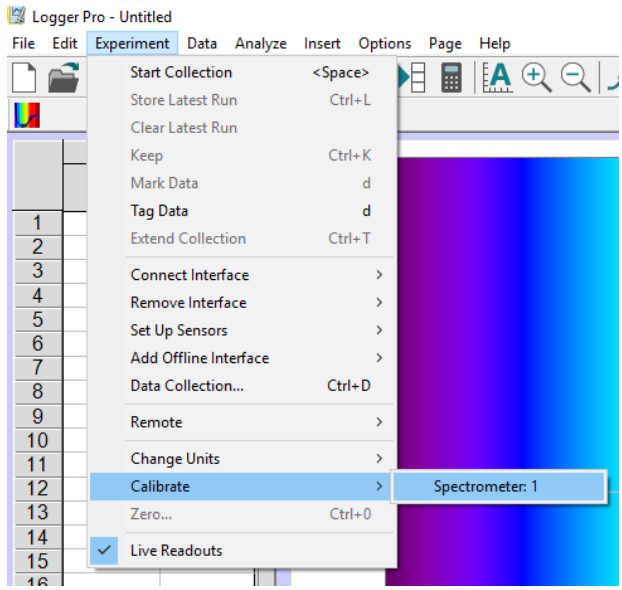


Fig. 2: Spectrometer Calibration

7. Fill one of the plastic cuvettes about three-fourths of the way full with the solution from Tube #1 (your blank). Cap it. Clean the smooth sides of the cuvette with a Chemwipe to remove fingerprints and dust, and then insert it into the sample holder in the spectrophotometer. Make sure that the smooth side is facing the light's path, indicated by the triangles on opposite sides of the sample holder.

8. Follow the instructions in the dialog box to complete the calibration and then press OK.

9. Clean and dry the cuvette. Fill it about three-fourths of the way full with the solution from Tube #2. Cap it. Clean the smooth sides of the cuvette with a Chemwipe and then insert it into the sample holder in the spectrophotometer.

10. Click Collect. A full spectrum will be recorded across 400-750 nm. Click Stop to end data collection.

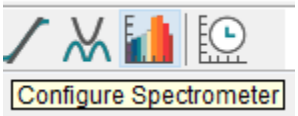
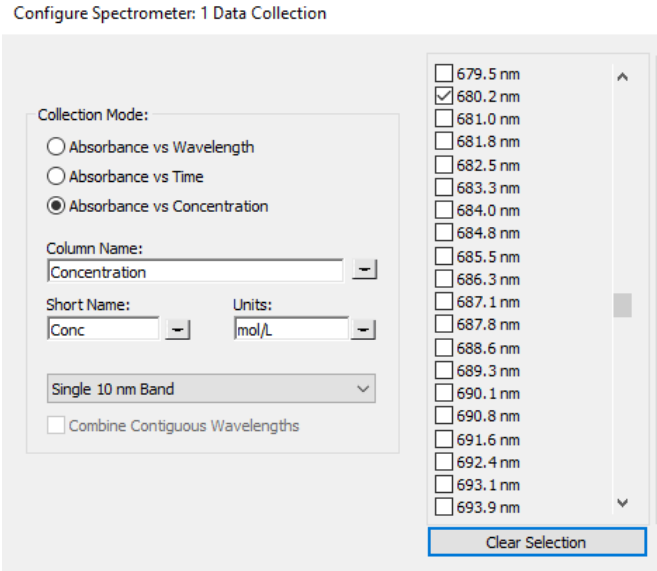


Fig. 3: Configure Spectrometer



11. Click the Configure Spectrometer Data Collection button (Fig. 3, 4). Select "Abs vs. Concentration" as the collection mode. The wavelength of maximum absorbance (λ_{max}) will be automatically selected from your previous graph from the curve's maximum. This number should be close to 680 nm. If it is not, click Clear and type in 680. Otherwise, click OK.

Fig. 4: Collection Mode, λ_{max}

12. With the solution from Tube #2 still in the sample holder, press Keep. When prompted, enter the concentration of the sample and click OK. Record the absorbance on your data sheet.

13. Dispose of the solution from the cuvette in the appropriate waste container.

14. Wash with deionized water and then thoroughly dry the cuvette before filling about three-fourths of the way full with the solution from Tube #3. Cap it. Clean the smooth sides of the cuvette with a Chemwipe and then insert it into the sample holder in the spectrophotometer.

15. Press Keep. When prompted, enter the concentration of the sample and click OK. Record the absorbance on your data sheet.

16. Repeat Step 12-14 with Tubes #4-6.

17. Click Stop to end data collection.

18. Click the Linear Fit button. The equation for the trendline will be automatically listed on the graph. Record this on your data sheet.

19. Print the graph and include it with your post-lab assignment.

Part III: Determination of Saturated Solutions

1. Obtain seven clean, dry small test tubes.

2. Add approximately 0.8-0.9 g of $CuSO_4 \cdot 5\ H_2O(s)$ crystals and 1.5 mL of deionized water to Tube #1.

3. Using a volumetric pipette, measure 2.50 mL of deionized water into Tubes #2-6.

4. Add approximately 3 mL of deionized water to Tube #7. This will be your "blank".

5. Prepare an ice bath by filling a large beaker approximately 2/3 full of ice and tap water.

6. Place Tube #1 in the ice bath in Step 5. Stir the ice-water mixture with a glass stirring rod while monitoring the temperature with a glass thermometer. When the temperature ceases to decrease, record it in your data sheet. Wipe the stirring rod dry before using it to stir the mixture inside Tube #1 for three minutes. Keep the tube in the ice bath and let it settle for at least one minute.

7. Using the same plastic pipette from Part I, carefully transfer exactly 10 drops from the top of the saturated solution in Tube #1 to Tube #2. Squirt any excess solution left in the pipette back into Tube #1.

8. Discard the ice bath in the sink and fill the beaker approximately two-thirds full of room temperature tap water. Put Tube #1 back into the water bath. Stir the water bath with a glass stirring rod for at least three minutes. Wipe the stirring rod dry before using it to stir the mixture inside Tube #1 for about three minutes. Record the temperature of the water bath on your data sheet.

9. Allow the mixture inside Tube #1 to settle for at least one minute. Using the same plastic pipette from Part I, transfer exactly 10 drops from the top of the saturated solution Tube #1 to Tube #3. Squirt any excess solution left in the pipette back into Tube #1.

10. Gather a Bunsen burner, iron ring, and wire gauze. Set the beaker with the water bath and Tube #1 carefully on top of the burner and iron ring.

11. Heat the water bath and Tube #1 to approximately 40°C. Stir the water bath with a glass stirring rod for at least three minutes. Wipe the stirring rod dry before using it to stir the mixture inside Tube #1 for about three minutes. Record the temperature of the water bath on your data sheet.

12. Allow the mixture inside Tube #1 to settle for at least one minute. Using the same plastic pipette from Part I, transfer exactly 10 drops from the top of the Tube #1 to Tube #4. Squirt any excess solution left in the pipette back into Tube #1.

13. Repeat Steps 11-12 twice more, heating the water bath to approximately 60°C and then 80°C.

> If you run out of liquid in Tube #1, add approximately 0.50 mL of deionized water.
> If you run out of crystals in Tube #1, add approximately 0.5 g $CuSO_4 \cdot 5\ H_2O(s)$.

14. Shake Tubes #2-6 gently.

15. Repeat Part I Steps 4-17 to set up the spectrophotometer. Use Tube #7 as your "blank" to calibrate the spectrometer. Use stock solution (0.200 M $CuSO_4 \cdot 5\ H_2O$) to find λ_{max}, which should still be around 680 nm.

16. Change the collection mode via the Configure Spectrometer button to Absorbance vs. Concentration. Record the absorbance for Tubes #2-6 on your data sheet. When asked for concentration, enter the tube number. Do not print this graph.

17. Dispose of Tubes #1-6 in the appropriate waste container.

18. Using the trendline equation from the graph in Part II, convert each absorbance to concentration, in M, for Tubes #2-6 for the dilute solutions.

19. Using $M_1V_1 = M_2V_2$, convert the dilute solution concentrations in Step 18 to the saturated solution concentrations from the original 10 drops removed from Tube #1.

20. Plot a graph of temperature (x-axis) versus concentration of saturated solutions (y-axis). Determine whether the relationship between solubility and temperature is direct or inverse. Print out the graph and include it with your post-lab assignment.

Experiment 21—Data Sheet

Name: _____

Part I: Calibration of the Plastic Pipette

Number of Drops	Total Mass (g)	Mass of 10 Drops (g)	Volume of 10 Drops (mL)
0			
10			
20			
30			

1. Temperature of deionized water (°C) _____

2. Density of water at this temperature from CRC (g/mL) _____
show calculations for one 10 drop portion:

3. Average volume of 10 drops (mL) _____
show calculation:

Part II: Determination of Standard Solutions

Tube #	mL of 0.200 M $CuSO_4 \cdot 5\ H_2O$ added	mL H_2O added	M $CuSO_4$ diluted	Absorbance
2	about 3	0	0.200	
3	4.00	1.00		
4	3.00	2.00		
5	2.00	3.00		
6	1.00	4.00		

show calculation for Tube #3 diluted concentration:

1. Trendline Equation from graph: _____

Part III: Determination of Saturated Solutions

Tube #	Temperature (°C)	Absorbance	Molarity (dilute)	Molarity (saturated)
2				
3				
4				
5				
6				

show calculation for Tube #2, Molarity (dilute) (use graph equation):

show calculation for Tube #2, Molarity (saturated):

1. Is the relationship between solubility and temperature direct or inverse? Explain.

Experiment 21—Post-Lab Assignment

1. Using your calculated molarity (saturated) from Tube #2 (ice bath), calculate the mass percent of the saturated $CuSO_4 \cdot 5\ H_2O$ solution. Assume the solution's density is 1.12 g/mL. (Hint: Assume 1 L of total solution)

2. Determine the grams of solute and grams of water present in 100 g of the solution in (1).

3. Calculate the grams of $CuSO_4 \cdot 5\ H_2O$ in 100 grams of water. This is the solubility.

4. Look up the CRC Handbook value for the solubility of $CuSO_4 \cdot 5\ H_2O$ in 100 g water at 0°C. Calculate the percent error for your answer in (3).

5. Give three experimental reasons why your value may be different than the CRC's.

Experiment 21—Pre-Lab Assignment

Name: _____

For all calculations, show all work and draw a box around the final answers.

1. Does the solubility of all solids in water increase with temperature? If not, list any exceptions shown in Figure 1 (do not include gases).

2. A 0.0500 M solution had an absorbance of 0.399. An unknown solution of the same solute in the same spectrometer had an absorbance of 0.231. Calculate the second solution's concentration, in molarity (Hint: recall that ε and l are constant).

3. 10 drops of a 0.450 M NaCl solution was added to 5.0 mL of water. Assuming that the volume of each drop is 0.050 mL, find the molarity of the dilute solution.

4. In Part III, why is it not important to know the exact amount of water and $CuSO_4 \cdot 5\ H_2O(s)$ in Tube #1?