

# Experiment 17

## SOLUBILITY OF IONS AND CUPRIC SULFATE PENTAHYDRATE

### Abstract

In this experiment, you will qualitatively measure the solubility of pairs of ionic compounds resulting from double displacement reactions between aqueous solutions. You will also determine and plot the solubility curve for cupric sulfate pentahydrate ( $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ ) using spectrophotometry and Beer's Law.

### Background

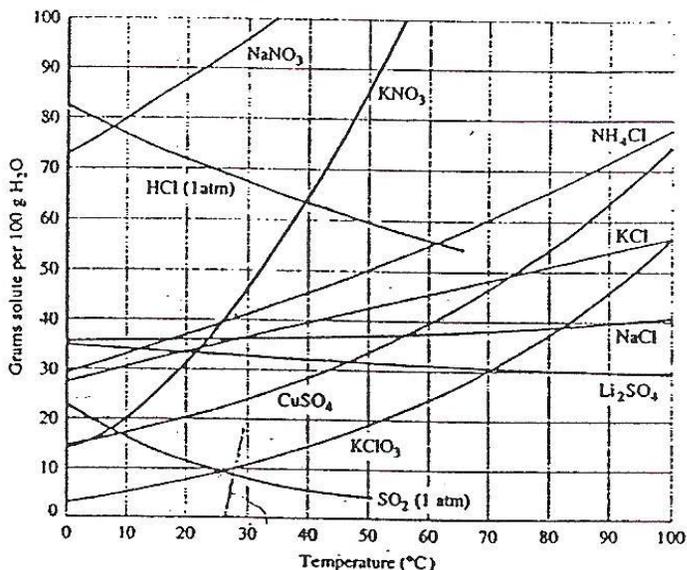
A *double displacement* reaction involves two sets of ionic compounds that switch partners according to the following generic equation (Eqn. 1):



Here, A and B represent cations and X and Y represent anions. If AX and BY are both aqueous solutions and either AY or BX are insoluble in water, the reaction can be further classified as a *precipitation reaction*, referring to the formation of a solid product from non-solid reactants. If neither AY nor BX remain as a solid in water, it can be said that no reaction has occurred as no change has taken place from reactants to products, and thus no net ionic equation will exist.

The formation of a precipitate is dependent on the solubility of the products. Compounds that remain solids in aqueous solution are classified as *insoluble*, whereas compounds that dissolve into separate ions in aqueous solution are classified as *soluble*. The degree of solubility of a compound in a particular solvent is dependent on a number of factors, including temperature, concentration, and pH.

Figure 1



In general, the solubility of solids increases with increasing temperature. Plotting the solubility of a compound against temperature is known as a *solubility curve*, as seen in Fig. 1.

The solubility of the compound can be measured in terms of concentration via spectrophotometry. A beam of light is shined onto a sample of the solution which absorbs some of its intensity while the rest passes through. The spectrophotometer measures the ratio of light passed through the sample to

the original intensity of the light source, giving readings in units of *percent transmittance* (%T). These values can be converted to the amount of light absorbed (absorbance, A) by the sample with Eqn. 2:

$$A = -\log(\%T/100) \quad (2)$$

Here, percent transmittance is converted to a decimal. The negative log of this value is taken to convert the reading into absorbance units. This value can then be related to the concentration of the solution via Beer's Law (Eqn. 3):

$$A = \epsilon l C \quad (3)$$

Here,  $\epsilon$  is the molar absorptivity constant (in  $M^{-1} \cdot cm^{-1}$ ) individual for the solution,  $l$  is the path length of the light source (usually the width of the sample container used in the spectrophotometer, in cm), and  $C$  is the concentration of the solution (in M). By Eqn. 3, the absorbance and concentration of a sample are directly related. Therefore, a plot of absorbance versus concentration should be linear with a constant slope as long as  $\epsilon$  and  $l$  are constant. This equation from the graph can be used to calculate the concentration of unknown samples of solution so long as their absorbance is known.

## Procedure

### SOLUBILITY OF IONS (You may do this part of the experiment with a partner)

1. Gather a plastic sheet covered mixing chart and bring it to your desktop. Avoid moving it during the experiment to prevent spilling.
2. Place one drop of 0.10 M of the solution listed on the mixing chart into each square on the plastic sheet protector in the column or row indicated on the chart. Place the drop in such a way that puts half of it in the black column and half in the white to ensure that you can see any precipitate form.
3. Continue adding drops of the appropriate solutions to each column or row until every square has had its two solutions mixed together.
4. Allow a few minutes to elapse after mixing as some precipitates may form slowly. Record your observations for each square in the appropriate spot on the report grid, writing "PPT" and indicating its color if a precipitate forms. If no precipitate forms, write "NR".
5. For each reaction observed, write its net ionic equation on a separate sheet of paper to attach to your report sheet.

### PREPARATION OF STANDARD SOLUTIONS

1. Obtain 6 identical test tubes that will fit into the spectrophotometer's sample holder. The tubes should be clean, dry, and free from scratches or discoloration. Number the tubes.
2. Fill the tubes with the following:
  - #1: about 3 mL of deionized water
  - #2: about 3 mL of 0.200 M  $CuSO_4(aq)$

**WARNING:** 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.

- Using a volumetric pipette to measure the volumes, fill the remaining tubes with the following:
  - #3: 4.00 mL of 0.200 M  $\text{CuSO}_4(\text{aq})$  + 1.00 mL deionized water
  - #4: 3.00 mL of 0.200 M  $\text{CuSO}_4(\text{aq})$  + 2.00 mL deionized water
  - #5: 2.00 mL of 0.200 M  $\text{CuSO}_4(\text{aq})$  + 3.00 mL deionized water
  - #6 1.00 mL of 0.200 M  $\text{CuSO}_4(\text{aq})$  + 4.00 mL deionized water

### DETERMINATION OF STANDARDIZED ABSORBITION

- Prepare the spectrophotometer by putting it into %T mode by pressing the “Mode” button.
- Set the wavelength to 680 nm by rotating the top right knob.
- With the sample holder empty and its door closed, adjust the reading to 0.00% T using the knob on the left front of the machine.
- Take Tube #1 and wipe the outside of each tube carefully to remove fingerprints and dust before inserting it into the spectrophotometer (do this for each subsequent sample). With Tube #1 in the spectrophotometer, adjust the %T reading to 100.0% using the right hand knob on the front of the machine.
- Remove the tube and close the sample door. Ensure that the %T still reads 0.0%. If not, readjust to 0.0% and then insert Tube #1 again to read 100.0%.
- Put the machine in Absorbance mode by pressing the “Mode” button.
- Wipe the outside of Tube #2 in the same fashion as Tube #1. Insert Tube #2 into the sample holder. Record the absorbance.
- Repeat Step 7 for Tubes #3-6.
- Dispose of the tubes’ contents into the specified waste container.

### CALIBRATION OF THE BERAL PIPETTE

- Weigh smallest Erlenmeyer flask and stopper available while empty. Record the mass.
- Fill a disposable beral pipette about  $\frac{1}{4}$  full of deionized water. Hold the dropper vertically and practice dispensing and counting single drops of liquid into a waste container.
- Once confident, dispense exactly 10 drops of deionized water into the empty Erlenmeyer flask and stopper it. Record the mass.
- Add exactly 10 more drops of deionized water to the Erlenmeyer flask and stopper it. Record the new mass.
- Add exactly 10 more drops of deionized water to the Erlenmeyer flask and stopper it. Record the third mass.
- Record the temperature of the sample of deionized water used.
- Squirt the pipette dry for later use.

### PREPARATION OF SATURATED SOLUTIONS

- Using the cleaned and dried six tubes from the standardized solutions, add 2.50 mL of deionized water via a volumetric pipette to each of the Tubes #2-6.

2. Add 0.8-0.9 g of copper(II) sulfate pentahydrate crystals and 1.2 mL of deionized water to Tube #1.
3. Place Tube #1 into an ice bath prepared by filling a beaker 2/3 full of an ice-water mixture. Stir the ice-water mixture with a glass stirring rod while monitoring the temperature. When the temperature ceases to decrease, record it. Wipe the stirring rod dry before using it to stir the contents of Tube #1 for 3 minutes. Keep the tube in the ice bath and allow it to settle for at least a minute.
4. Using the calibrated beral pipette, 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.
5. Discard the ice bath and fill the beaker with about 2/3 full of room temperature tap water. Put Tube #1 back into the water bath. Stir the bath with a glass stirring rod first before stirring the solution in Tube #1 for about 3 minutes. Allow it to settle for at least 1 minute. Record the temperature of the water bath.
6. Using the calibrated beral pipette, carefully 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.
7. Heat the water bath to about 40°C, repeating the process of stirring, recording the temperature, and transferring 10 drops from Tube #1 to Tube #4.
8. Increase the temperature of the water bath to about 60°C and then 80°C to fill Tubes #5 and #6 respectively, following the same process of stirring, recording the temperature, and transferring 10 drops.
  - If you run out of liquid in Tube #1, add about 0.50 mL of deionized water.
  - If you run out of crystals in Tube #1, add about 0.5 g  $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ .
9. Before measuring the absorbance of Tubes #2-6, stopper and shake gently. Wipe the outside of the tube clean before inserting into the spectrophotometer. Record the absorbance of each.

### DATA ANALYSIS

1. Calculate the molarities of your standardized solutions using  $M_1V_1 = M_2V_2$ .
2. Using the data collected from your standardized solutions of  $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ , prepare a graph of molarity versus absorbance. Record the equation generated from the trendline.
3. Use this equation to determine the molarity of each of the five dilute solutions prepared from the original saturated solution from their recorded absorbances.
4. Use these concentrations to calculate the concentration of the original saturated solution of the ten drops taken from Tube #1 before dilution with 2.5 mL of water at each of the 5 temperatures using  $M_1V_1 = M_2V_2$ .
5. Plot these concentrations (molar solubility) versus temperature on a second graph.

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## Report Sheet

**Solubility of Ions:** Record observations in each square. Precipitate = “ppt” (indicate color), No reaction = “NR”.

**Horizontal Ions:** The cations are spectators. **Vertical:** The anions are spectators.

	$\text{NaC}_2\text{H}_3\text{O}_2$	$\text{KBr}$	$\text{Na}_2\text{CO}_3$	$\text{KCl}$	$\text{K}_2\text{CrO}_4$	$\text{Na}_3\text{PO}_4$	$\text{Na}_2\text{SO}_4$	$\text{NaOH}$	$\text{Na}_2\text{S}$	$\text{KNO}_3$	$\text{K}_2\text{C}_2\text{O}_4$
$\text{NH}_4\text{Cl}$											
$\text{BaCl}_2$											
$\text{CaCl}_2$											
$\text{Cu}(\text{NO}_3)_2$											
$\text{FeCl}_3$											
$\text{MgCl}_2$											
$\text{KNO}_3$											
$\text{AgNO}_3$											
$\text{Zn}(\text{NO}_3)_2$											
$\text{Al}(\text{NO}_3)_3$											

### Summary of Solubility of Ions:

1. List the ions that formed no precipitates.
2. List the ions that formed precipitates with all ions except those listed in Q1.
3. What is the general relationship between the solubility of ions and their charge?
4. Develop a set of general solubility rules based on the observations listed above.

### Standardized Solutions

Tube #	mL of 0.200 M CuSO <sub>4</sub>	mL H <sub>2</sub> O	M CuSO <sub>4</sub>	Absorbance
2	~ 3	0	0.200 M	
3	4.00	1.00		
4	3.00	2.00		
5	2.00	3.00		
6	1.00	4.00		

Calculate the diluted concentration for Tubes #3-6 (show 1 calculation setup):

### Beral Pipette Calibration

Number of Drops	Mass	Mass of 10 drops	Volume of 10 drops
0		-----	-----
10			
20			
30			
<b>Average</b>	-----	-----	

Density of water at this temperature from the CRC \_\_\_\_\_

Calculate the volume of each increment using the density of water (show 1 calculation setup):

### Saturated Solutions

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

Calculate the molarity of each of the dilute solutions (show 1 calculation setup).

Calculate the molarity of each of the saturated solutions (show 1 calculation setup).

### Post-Lab Assignment – Experiment 17

1. Calculate the mass percent of the saturated  $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$  solution when in the ice bath, assuming the solution's density is 1.12 g/mL. (Hint: Assume 1 L of total solution and use the solution's calculated molarity)

2. Determine the grams of solute and the grams of water present in 100 g of the above solution.

3. Calculate the solubility of  $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$  at  $0^\circ\text{C}$  in grams of solute per 100 grams water.

4. What is the CRC Handbook value for this? \_\_\_\_\_

5. Assuming the above value is correct, calculate your percent error.

NAME: \_\_\_\_\_

**Pre-Lab Assignment – Experiment 17**

1. Does the solubility of all solids in water increase with temperature? If no, 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 had an absorbance of 0.231. Calculate the second solution's molarity.

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. When  $\text{AgNO}_3(\text{aq})$  is added to  $\text{Na}_3\text{AsO}_4(\text{aq})$ , a brown precipitate results.

a. Write the balanced molecular equation for this reaction.

b. Write a total ionic equation for this reaction.

c. Write a net ionic equation for the reaction.