Solutions and Spectroscopy

PURPOSE

To determine the concentration of a copper sulfate ($CuSO_4$) solution, and to duplicate its concentration by two methods.

GOALS

- To learn how to use a pipet properly.
- To learn how to dilute a stock solution.
- To learn how to use a spectrophotometer.
- To gain practice plotting a calibration curve and use it to determine the concentration of an unknown solution.
- To learn how to make a solution from a solid reagent.
- To learn how to make a solution by diluting a stock solution.

INTRODUCTION

A solution is a homogeneous mixture of two or more substances. Simple solutions consist of one solvent and one or more solutes. The solvent is the major liquid component of the mixture in solutions that contain one or more liquids. The most common solutions are aqueous solutions, in which water is the solvent. The concentration of a solute is the ratio of the amount of solute to the amount of solution or solvent. One of the most common ways to report concentration is in units of **molarity**[1]. Molarity is defined as the number of moles of solute in one liter of solution. It has units of **moles/liter** (mol/L) and is given the symbol **M**. For example, if 1.5 moles of sodium chloride is dissolved in enough water to make 1.0 liter of solution, the concentration of sodium chloride is 1.5 M, or 1.5 mol NaCl/L of solution. This would be spoken as "1.5 molar."

In order to accurately make solutions of known molarities, we must accurately determine the number of moles of the solute *and* the volume of the solution. Several pieces of equipment have been developed to measure accurate volumes. These include volumetric flasks, burets, pipets, and graduated cylinders. Other glassware, such as beakers and Erlenmeyer flasks, have volume markings on them, but they are less accurate.

Most general chemistry students are familiar with the graduated cylinder. It is reasonably accurate and precise (±0.5 to 1 mL depending on its size) and can be used for many measurements. However, greater accuracy and precision is often needed. Several simple devices accurate to 0.01 mL or better are in common use in laboratories. These are:

1 **Volumetric flasks**. These pear-shaped flasks are designed to contain a volume of liquid or solution which is known to 0.1 mL or better. They have a narrow neck with a single mark. Solvent is added to the mark.

[1] http://en.wikipedia.org/wiki/Molar_concentration

2 **Burets**. These long, narrow, graduated tubes have a stopcock at the bottom. They are designed to dispense variable amounts of liquid or solution. The volume of solution in the buret is recorded before dispensing the solution and afterward. The difference between the two readings is the volume of liquid dispensed. The volume readings should be to two decimal places. Burets are most often used in titrations.

3 **Pipets**. Pipets, like burets, are designed to dispense a known volume of solution. There are several common types:

a Volumetric, or transfer, pipets have a single mark on a narrow neck. They are the most accurate pipets for delivering a known amount of liquid or solution. They are drained by gravity. Remaining traces of liquid are not forced out, e.g. by blowing on the pipet.

b Mohr pipets, or measuring pipets, have graduations and are used much like burets. They are not drained completely.

c Serological pipets are graduated and are designed to be drained completely. Most are designed to have the last traces of liquid blown out.

If the concentration of a solution is unknown, the concentration can be measured by determining the amount of light it absorbs (its absorbance, A) at a particular wavelength (λ), using a spectrophotometer. Absorbance and concentration, c, are directly related by Beer's Law:

$$\mathrm{A} = \epsilon\, l\, \mathrm{c} \tag{1}$$

where ϵ is the "molar absorptivity" (a constant unique to that solute at that wavelength) and l is the path length, or distance the light travels through the solution.

To evaluate c from absorbance measurements, one measures the absorbance of several *standard solutions* (solutions of known concentration). A plot of A vs. c, which equation 1 shows should be linear, allows calculation of ϵ. In these measurements, l is held constant by using the same sample holder (or a carefully matched set of them) for all measurements.

The plot of A vs. c is called a calibration curve. The concentration of any other solution can be found with the calibration curve by measuring its absorbance.

There are two standard ways to make solutions of a desired concentration. First, if the solute is a solid, the appropriate amount of solid may be weighed out and then dissolved in enough solvent to make the desired amount of solution. This is often done in a volumetric flask. The accuracy and precision of the mass and volume determinations dictate the number of significant figures that can be used in reporting concentration. Usually, four significant figures are used.

The second common way to make a solution is to dilute a stock solution with additional solvent. The relationship between the concentrations and volumes of the concentrated and diluted solutions is,

$$\mathrm{C_i V_i} = \mathrm{C_f V_f} \tag{2}$$

where C_i and V_i are the concentration and volume of the initial (concentrated) solution and C_f and V_f are the concentration and volume of the final (diluted) solution.

The accuracy with which you measure volumes determines the number of significant figures you can use in reporting your concentration. Any error in the concentration of the stock solution (C_i) will be propagated into the concentration of the dilute solution (C_f).

For this experiment, assume that you are working at a company that uses copper(II) ion solutions in its processing. The production manager has sent you a small sample of the copper(II) ion solution they use and told you to make more at that specific concentration. Unfortunately, the production manager did not tell you what the concentration was, and she just left for vacation. Your job is to determine the concentration of the solution, and to generate more solution of that same concentration.

In Part A of this experiment, you will make several solutions with known concentrations of copper(II) ions, then make absorbance measurements on them, and develop a calibration curve. You will then measure the absorbance of the unknown solution and determine its concentration from that calibration curve.

In Part B, you will make a copper(II) ion solution with a concentration identical to the unknown by dissolving solid copper sulfate, $CuSO_4 \cdot 5\ H_2O$, in water.

In Part C, you will make a copper(II) ion solution with a concentration identical to the unknown by diluting a concentrated, stock copper sulfate, $CuSO_4$, solution.

You will compare the accuracy with which you can make the solutions by the two methods.

EQUIPMENT

1 MicroLab Spectrophotometer

1 MicroLab Spectrophotometer Instruction Sheet

6 vials

2 Serological pipets

1 pipet bulb

5 13 × 100 mm test tubes

5 stoppers

1 test tube rack

2 30 mL beakers

2 25 mL volumetric flasks w/ stoppers

1 250 mL beaker for copper waste

2 eye droppers

REAGENTS

25 mL 0.5XX M $CuSO_4(aq)$ stock solution

10 mL $CuSO_4(aq)$ unknown solution

1 g solid $CuSO_4 \cdot 5\ H_2O$

SAFETY

$CuSO_4 \cdot 5\ H_2O$ is listed as toxic and an irritant. Toxic substances are hazardous to health when breathed, swallowed or in contact with the skin. An irritant may have a temporary irritating effect on skin, eyes, respiratory tract, etc. If you come in contact with the solid, you should gently brush off the affected area with a paper towel and then flush the area with water. If you come in contact with a solution of copper(II) sulfate, you should flush the affected area with water.

WASTE DISPOSAL

Solutions containing copper ions must be placed in the waste bottle in the lab. Designate a "waste copper" beaker and set it aside for use during your lab. You can put the small samples of copper solution you will make in this and empty it into the waste bottle at the end of class, instead of going back and forth to the waste bottle. Always remember not to overfill the waste bottle. If your waste bottle is full, please alert your lab instructor.

PRIOR TO CLASS

Please read the following section of Lab Safety and Practices:

- Good Lab Practices
- Measurements
- Preparing Graphs

Please read the following section in Lab Equipment:

- Volumetric Glassware
- Analytical Balance

Please review the following videos:

- Using an Analytical Balance
- Pipeting Techniques
- Using a Volumetric Flask
- Laboratory Safety

LAB PROCEDURE

Please print the worksheet for this lab. You will need this sheet to record your data.

Part A: Determination of the Concentration of a Copper(II) Ion Solution

1 A spectrophotometer will be set up in your work area. Make sure it is turned on and allow it to warm up.

2 While the spectrophotometer is warming up, obtain two serological pipets, and set up a beaker for waste Cu^{2+} solution.

3 Obtain about 15 mL stock Cu^{2+} solution. Write its exact concentration in Data Table A.

4 Once the spectrophotometer is warmed up, take a blank spectrum of deionized water in a vial as described in the MicroLab spectrophotometer instructions provided in lab.

5 Condition the pipet you will be using for the Cu^{2+} solution as shown in the pipet video under instrumentation and as described in the Volumetric Glassware section of the Introductory Material of this lab manual. Dispense the required volumes of Cu^{2+} solution (1.2 mL) and deionized water (4.8 mL) for Solution 1 into a test tube and record your volumes in Data Table A.

- Be careful while doing this, the pipets may be tricky to handle at first.
- Record volumes to 0.01 mL with some uncertainty in the last digit.

6 Stopper the test tube and mix the solution well by inverting a couple of times.

7 To condition your vial, carefully pour a small amount of the solution you just prepared into a vial and pour it out to waste.

8 Refill the vial with solution and take an absorbance spectrum. Identify the wavelength of maximum absorbance near 600 nm. Record the wavelength and absorbance at this wavelength in Data Table A. Absorbance values are reported to the 0.001. For all remaining absorbance measurements in this experiment, be sure to use the same wavelength you have identified as the maximum.

9 Record the concentration of Cu^{2+} in Solution 1 in Data Table A.

10 Retain the sample in the vial until you have completed your calibration plot. Students often choose to label a sheet of paper with positions 1, 2, 3 and 4, placing each vial on the appropriate position.

11 Following steps 5 - 10, prepare Solutions 2 - 4, measure their absorbances and calculate the Cu^{2+} concentration in each. Record these in Data Table A. Do not close the MicroLab file, as this calibration curve will be used to determine the concentrations in Part B.

12 The MicroLab software will plot the absorbance of the Cu^{2+} solutions that you made as a function of their concentrations. The trendline and R^2 value are displayed. If your plot is linear with an R^2 value of 0.9 or greater, continue the experiment. If your R^2 value is low, consult with your lab instructor.

13 Safely dispose of the calibration solutions in your copper waste beaker.

14 Obtain a couple of milliliters of unknown solution and record the number of your solution in Data Table A.

15 Condition a vial with the unknown Cu^{2+} solution and refill with unknown Cu^{2+} solution. Measure the absorbance of the unknown Cu^{2+} solution and record it in Data Table A.

Part B: Preparation of a Copper(II) Ion Solution from Solid $CuSO_4 \cdot 5\ H_2O$

1 Carefully weigh the desired amount of $CuSO_4 \cdot 5\ H_2O$ on a weighing paper or in a small beaker as shown in the Using an Analytical Balance video under Instructional Videos and as described in the Analytical Balance section of the Lab Equipment of this lab manual. Record the exact amount that you used in Data Table B.

2 Transfer the $CuSO_4 \cdot 5\ H_2O$ into a 25.00 mL volumetric flask and dilute to the mark with deionized water as shown in the Volumetric Flask video under Instrumentation and described in the Volumetric Glassware section of in the Introductory Material of this lab manual.

3 After the solution is well mixed, condition a vial with it. Then refill the vial, measure and record the absorbance of your solution nm in Data Table B.

Part C: Preparation of a Copper(II) Ion Solution by Dilution of a Stock $CuSO_4$ Solution.

1 Record the exact concentration of the stock copper(II) solution in Data Table C.

2 In a small, clean beaker, obtain a little more of the stock copper(II) solution than you calculated in Question 6.

3 First condition a serological pipet, then use it to transfer the volume of copper(II) solution you calculated into a 25.00 mL volumetric flask. Record the exact amount that you transferred in Data Table C. Dilute to the mark with deionized water.

4 After the solution is well mixed, condition a vial with it. Then refill the vial, measure and record the absorbance of your solution in Data Table C.

5 When you are finished taking measurements, collect all your copper solution waste and place it in the waste bottle in the lab, making sure not to overfill it. Rinse and dry all your glassware with water and return it to the set-up area where you found it. Close the MicroLab software.

6 Remember to show your TA your calibration curve and measured $[Cu^{2+}]$ concentration of your solutions for Part B and Part C. Your TA will manually grade the results and enter your score into WebAssign.

7 Before leaving, enter your results in the In-Lab assignment. If all results are scored as correct, log out. If not all results are correct, try to find the error or consult with your lab instructor. When all results are correct, note them and log out of WebAssign. The In-Lab assignment must be completed by the end of the lab period. If additional time is required, please consult with your lab instructor.