# Experiment 1

# REVIEW OF LAB TECHNIQUES

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## PURPOSE

To review density calculations, Beer's Law and the use of electronic balances, volumetric glassware, and spectrometers.

### BEER'S LAW

Colored species in solution absorb visible light. As the amount (concentration) of the species increases, so will the amount of light it absorbs. The fraction of incident light that passes through a solution, without being absorbed, is called the **transmittance *T***, which is defined as

$$T = I/I_0 \qquad (1)$$

where $I$ is the intensity of light emerging from the solution and $I_0$ is the intensity of light that entered the solution. **Percent transmittance %*T*** is the percent equivalent of the fraction *T*:

$$\%T = I/I_0 \; x\, 100\% \qquad (2)$$

If all of the light is absorbed then %*T* = 0. If all of the light is transmitted then %*T* = 100.

Although %T decreases as the concentration of a colored species increases, the relationship is not linear. However, the concentration *is* a linear function of the *logarithm* of %*T*. Therefore, a quantity which is linear in concentration, called the **absorbance** *A,* has been defined:

$$A = 2 - \log \%T \qquad (3)$$

This definition, utilizing common logs, is a relic from the days before electronic calculators when logarithms were obtained from "log tables." It is a convenient choice since A = 0 when %T=100.

The equation describing how the absorbance *A* depends on concentration is referred to as **Beer's law** (although Mr. Beer was not solely responsible for it):

$$A = \varepsilon bc \qquad (4)$$

where $\varepsilon$ is the **molar absorptivity** or the **molar extinction coefficient**, *b* is the path length (the distance the light travels through the solution), and *c* is the molar concentration of the absorbing species.

The molar absorptivity $\varepsilon$ is different at each wavelength. It will have its maximum value at the wavelength of light most efficiently absorbed by the colored species. Therefore, because the absorbance A is unitless (which should be clear from its definition, see eq. 3), the units of $\varepsilon$ must be the reciprocal of the units of *b* times *c*. $\varepsilon$ can be determined by measuring the absorbance for a solution of known b and *c*. The concentration of an unknown solution can then be determined by measuring *A* and solving Beer's law for *c*.

### IN THIS EXPERIMENT

You will prepare a 0.0500 *M* $CuSO_4$(aq) solution and measure its density. The absorbance of this solution will be measured at 680 nm and 710 nm to determine its molar absorptivity at those wavelengths (demonstrating that $\varepsilon$ changes with wavelength). The concentration of an unknown solution of $CuSO_4$(aq) will then be determined by measuring its absorbance and using Beer's law.

# PRE-LABORATORY PREPARATION

**1. Read the background, procedure and data analysis sections of the experiment.**
**2. Review the use of volumetric glassware (pipets and volumetric flasks) and density calculations.**
**3. Answer the question on Beer's Law in the Data Analysis section.**

# EXPERIMENTAL SECTION

## REAGENTS PROVIDED

**0.500 *M* $CuSO_4$.**
**Unknown $CuSO_4$ solution.**

### Hazardous Chemicals

$Cu^{2+}$ is somewhat toxic. The oral $LD_{50}$ for rats is 300 mg/kg. A 1 kg rat has a 50% chance of dying if it drinks 10 mL of the 0.500 *M* solution.

## WASTE DISPOSAL

Discard $Cu^{2+}$ solutions in the designated bottles.

## PROCEDURE

**PREPARATION OF 0.0500 *M* $CuSO_4$**

You will do this part individually.

**1. Practice using a 10-mL pipet by filling it with distilled water and transferring the water to a 250-mL beaker.**

Repeat until you feel confident of your ability to use a pipet accurately.

**2. Rinse your pipet.**

Take about 30 mL of the 0.500 *M* $CuSO_4$ in a clean, dry 100-mL beaker. *Do not take more than you need.* Rinse your pipet twice with two small portions of the $CuSO_4$ solution (slightly more than a pipet stem full). Discard the rinsings in a 250-mL beaker which you should use as your temporary waste solution container.

**3. Pipet 10 mL of the $CuSO_4$ into a 100 mL volumetric flask. Fill to the mark with distilled water, cap the flask and invert several times to mix.**

*The density and spectrophotometric studies can be done in either order. Begin by doing the study for which you can most readily obtain access to the necessary equipment.*

**DETERMINATION OF DENSITY**

You will do this part individually.

**4. Rinse your pipet with the 0.0500 *M* $CuSO_4$ solution that you prepared.**

Discard your rinsings as before.

**5. Place a clean, dry 50-mL Erlenmeyer flask on an electronic balance, tare (zero) the balance, and then transfer 10.00 mL of the 0.0500 *M* $CuSO_4$ into the flask.**

This transfer should be done while the flask is on the table top, *not while it is on the balance pan.* This is to avoid the possibility of spilling chemicals on the balance.

**6. Record on your Data Sheet (in ink) the mass of the solution transferred.**

Always record your data readings as precisely as possible. So, record the mass to the nearest 0.001 g. If the last digit is fluctuating (which often occurs) record what you estimate is its average value.

**7. Tare the balance again (while the flask is still on the balance), remove the flask, and then add a second 10-mL aliquot to the flask. Place the flask back on the balance and record the mass.**

If, in your judgment, the readings are too different, repeat until you have two reasonably consistent values.

**SPECTROPHOTOMETRIC ANALYSIS**

This part may be done with a partner.

**8. Turn on the LabQuest2 by pressing the red power button on the top.**

It takes an honest minute to warm up.

**9. Connect the cable from the SpectroVis spectrometer to the USB port on the LabQuest2.**

The USB port is on the left side of the Lab Quest2 near the top. The screen should read "USB: Abs".

Note the words spectrophotometer and spectrometer are used interchangeably.

**10. Calibrate the spectrometer.**

a. Tap on ***Sensors*** from the menu line at the top of the screen. Next choose ***Calibrate*** and then ***USB: Spectrometer***.
b. Allow the spectrometer to go through the 90 second warm up if you have just connected it. (Otherwise, you may skip the warm up.)
c. Rinse a cuvet with distilled water three times. Then fill it about 3/4 the way with distilled water. This is called the *blank cuvet* (or cuvette). Insert the blank cuvet into the spectrometer. The clear sides of the cuvet should be facing left & right (in the direction of the ► on the spectrometer).
d. Tap ***Finish Calibration***. The calibration will finish in a couple of seconds. Then tap ***OK***.

**11. Obtain the absorbance spectrum (A vs. λ) of the 0.0500 *M* $CuSO_4$ solution you prepared.**

a. Rinse a cuvet with your solution and discard the rinse solution into a temporary waste beaker. Repeat for a total of three rinses. Fill your sample cuvet about 3/4 the way to the top. The outside of the cuvet should be carefully dried with a Kimwipe.
b. Remove the blank cuvet and insert the sample cuvet into the spectrometer. As always, the clear sides must be oriented so that the light beam goes through them. The direction of the light beam is indicated by the ► on the spectrometer.
c. By default, LabQuest2 starts in *Full Spectrum mode*. If you have changed to a different mode, you can change back by tapping on the Mode, selecting ***Full Spectrum*** from the box near the top right of the meter screen and then tapping on ***OK***.
d. Tap the Start button (with the Green arrowhead) in the lower level corner of the screen. If you have previously collected any data, you will be given the opportunity to save that data before it is erased. If you do not want it saved, simply tap on ***Discard***.
e. The spectrum should be displayed almost instantly. Wait a few seconds for it to roughly stabilize and then tap on the **Stop** button (with the red square) located where the Start button had been.
f. Tap on the **Data Table** icon (the white icon with the X|Y columns) found in the upper right corner of the screen. Using the scroll bar, as necessary, to see your data and *record the absorbances at 680 nm and at 710 nm* (or the closest wavelengths to them, if your spectrometer does not take readings at those exact wavelengths).

Typically, absorbances are displayed to the nearest 0.001 and should be recorded as such.

**12. Print your graph.**

a. Tap on the **Graph icon** (in the upper left corner of the screen) to see your graph again.
b. Tap ***File*** from the top menu.
c. Tap ***Print*** and then ***Graph***.
d. Tap on ***Print Graph Title***.
e. Enter an appropriate graph title. Usually, the title tells what is graphed in the format "Y" vs "X" (e.g., Absorbance vs Wavelength).
f. Tap ***Print***.

**13. Obtain about 20 mL of an unknown $CuSO_4$ solution in a clean, small beaker (50- or 100-mL). Rinse a cuvet with the unknown, fill the cuvet (3/4 of the way to the top), and then measure its absorbance spectrum, as in Step 11.**

Again, *record the absorbances at 680 nm and at 710 nm*. Also, *record the unknown number*.

You do not need to print this graph.

**14. Once you and your partner have both measured your unknown absorbances, shut down the LabQuest2.**

This can be done by first tapping ***File****, then* ***Quit***. Choose to ***Discard*** the data. Next, tap on the ***System*** folder and then ***Shut Down*** and, finally, ***OK***.

**15. Wash your glassware and put it away.**

Discard all of the $CuSO_4$ solutions into an appropriate waste bottle in the hood.

Do NOT dry the inside of the cuvets (or volumetric flasks) with paper towels. Shake them out, over a sink, a couple of times to remove most of the water (being careful not to spray the person standing next to you).

**RETURN EVERYTHING TO WHERE IT WAS AT THE START OF LAB. HAVE AN INSTRUCTOR CHECK YOUR STATION BEFORE LEAVING.**

**It is always a good idea to wash your hands before leaving lab.**

Name _____ Station Used _____ Instructor/Day/Time _____

Station Checked & Approved _____

# DATA SHEET

Record all values with the proper units and number of decimal places.

Mass of 10.00 mL aliquots of 0.0500 M $CuSO_4$ (take as many, or as few, readings as needed). Put a star (*) next to the good trials that you will use to calculate the density.

_____

_____

_____

_____

_____

| | Absorbance | |
|---|---|---|
| | 680 nm | 710 nm |
| 0.0500 *M* $CuSO_4$: | _____ | _____ |
| unknown $CuSO_4$: | _____ | _____ |

Unknown number: _____

# DATA ANALYSIS

Show ONE sample calculation for each problem. Each calculation should have the general formula, the formula with the numbers and units plugged in, and the answer *with proper units and significant figures*.

**1. Calculate the density of the 0.0500 *M* $CuSO_4$.** Assume that the volume of your pipet is 10.00 mL. Record the density for each good trial in the lines below. You should have a minimum of two good trials.

Density of Good Trial 1 = _____

Density of Good Trial 2 = _____

Density of Good Trial 3 = _____

Density of Good Trial 4 = _____

Average Density = _____

**2. Write Beer's Law and identify each term in it (on both sides of the equation).**

**3. Calculate the molar absorptivity of $CuSO_4$ at both 680 nm and at 710 nm, using the measured absorbances of the 0.0500 M solution that you prepared.** (The path length of the cuvets is 1.00 cm.)

$\varepsilon_{680}$ = _____

$\varepsilon_{710}$ = _____

**4. Using the measured absorbance of your unknown at 710 nm and the calculated molar absorptivity of $CuSO_4$ at 710 nm, determine the concentration of your unknown $CuSO_4$ solution.**

$[CuSO_4]$ = _____

Unknown Number = _____