MEASUREMENT: PART II

INTRODUCTION

Read and/or review Section 1.7 and Figure 7.5 in your textbook.

The first part of this week's lab deals with the measurement of volume.

The second part of the experiment deals with density. Density can be obtained from mass and volume measurements. The density of a liquid can also be determined with a hydrometer. A hydrometer is a graduated, weighted tube that sinks in a liquid to a point determined by the density of the liquid.

In the final part of the experiment, you will make solutions of a food dye and check their concentrations with a spectrometer. A spectrometer is an instrument that can be used to determine the concentration of a colored solution by analyzing the intensity of the color. The spectrometer measures the amount of light transmitted (T) through the solution. However, when determining concentration, the amount of light that is absorbed (A) by the solution is plotted as a function of concentration. The two are related by the equation:

$$A = \log \frac{1}{T}$$

You will not need to do the calculation that converts transmittance to absorbance. The spectrometer will do it for you.

The absorbance of a solution is proportional to its concentration. For example, a 50% solution will absorb half as much light as a 100% solution (in this experiment the food dye), a 25% solution will absorb one quarter as much light as a 100% solution, etc. For this experiment, all concentrations will be expressed as a percent by volume $\left[\%\,v/v = \left(\frac{\text{mL dye}}{\text{total mL solution}}\right) \text{ x } 100\%\right]$. This means that 100.0 mL of a 20.0% dye solution contains 20.0 mL dye and 80.0 mL water and 30.00 mL of a 15.0% dye solution contains 4.50 mL of dye and 25.50 mL of water.

PROCEDURE

A final reminder:

> Throughout the semester, make written observations as you proceed with the experiments. Be aware that there may be questions at the end of the lab that depend on observations.
>
> There is a general understanding about reading a value from a graduated device. Estimate the value to one decimal place more than the level of graduation. For example, when reading a thermometer graduated in degrees, estimate to tenths of a degree. For a ruler that is graduated in cm, estimate to mm.

VOLUME

Fill a wash bottle with deionized water (DI water) from the large container in the lab. You will need your smallest graduated beaker, a 25 mL graduated cylinder, a 10 mL pipet, and a 25 mL buret. Examine the imprinting or engraving on each of these volume measuring devices. If the measuring device is marked TD (or has a double band) it means that the device is made *to deliver* the stated volume and you should not force the release of any liquid that remains in the device. TC means *to contain* the stated volume and all contents in the device are included in the stated volume.

Determine the mass of 10 mL of DI water by dispensing measured volumes of water (using each glass device) into a tared plastic cup. Repeat this at least three times and then calculate an average mass.

DENSITY

Learn how to use a hydrometer. Use a hydrometer to find the density of water and the density of a salt solution. Pipet 10 mL of the same salt solution into a tared plastic dish or tray. Determine its mass. Calculate the density of the salt solution.

A CALIBRATION CURVE

Often, solutions of low concentration are best made by diluting a more concentrated solution (a stock solution) of the same reagent. You and your partner will prepare four solutions with assigned concentrations. To get your unknown assignment, go to the Laboratory page of our course web site that is set up on your lab computer. Enter in the appropriate information and print out the assigned concentrations.

For convenience, you will use a stock solution of a food dye in water to which we have assigned a concentration of 100.0%. The true concentration doesn't matter in this experiment. It is assumed that volumes are additive.

1. Double click the "Logger Pro" icon and allow the screen to open.

2. The spectrometer needs to be powered for about 5 minutes before using so do this step before preparing your solutions. Do not use the Go!Link with the spectrometer. Plug the spectrometer via provided USB cable to the computer USB port.

3. Pump dispense 35 mL of the stock dye solution into a small beaker.

4. Calculate the volume of stock solution that you must use to prepare 10.0 mL of each assigned solution. For example, to prepare 10.0 mL of a 15% solution, you need 1.5 mL of the stock solution since 15% of 10.0 is 1.5 (0.15 x 10.0 = 1.5).

5. Beginning with the most dilute solution (lowest concentration) assigned, pipet the calculated volume of stock solution into a 10 mL volumetric flask. Dilute to volume with water. Mix well and transfer into a labeled TT. Rinse the volumetric flask well and use it to make the next dilute solution. Repeat until all assigned solutions have been prepared.

6. Calibrate the Spectrometer ; Do not unplug the spectrometer during this experiment or you will have to start over.

    a. Return to the Logger Pro screen on the computer. Click: Experiment ; Calibrate ; Spectrometer:1.

    b. Allow the lamp to warm up for 90 seconds as displayed on the computer screen.

    c. Only touch the ridged faces of the cuvette, never touch the clear faces. Rinse and fill one cuvette (a cuvette should be filled so that it is about ¾ full) with the blank. In this lab the blank is deionized water. Rinse and fill (about ¾ full) a second cuvette with the most dilute of the solutions. **Gently blot** (don't scratch the sides of the cuvette) off any drips on the outside of the cuvette with a Kimwipe.

    d. Place the cuvette containing the blank in the spectrometer so that one of the clear sides is aligned with the white arrow at the top of the cuvette slot. Click: "Finish Calibration" ; OK.

7. Determine the Wavelength of Maximum Absorbance

    a. Place the cuvette containing the most dilute solution in the spectrometer.
    Click: Experiment ; Data Collection ; Full Spectrum ; Done. Click the rainbow icon labeled "Absorbance=…" in the upper left hand corner of the window. Change the "Wavelength Range" to 380-750nm. Close this box by clicking the "x" in the upper right hand corner of the window.

    b. Click the small green triangle in the toolbar labeled "Collect". After the line graph appears on the screen, click the small red square in the toolbar labeled "Stop".

    c. To automatically store the maximum wavelength go to the toolbar and select: Experiment ; Store Latest Run.

8. Generate the Calibration Curve. The graph (Absorbance vs. Concentration) generated should be linear and is called a calibration curve. If the four points fall on a straight line, your data are 100% correlated.

    a. In the toolbar click: Experiment ; Data Collection ; choose Events with Entry in the Mode box. Highlight the word Event in the Column Name box and replace it with Concentration. Put percent in the Units box. Clear "Short Name". OK. You are ready to begin collecting data. Remove the cuvette from the spectrometer.

    b. Place the cuvette containing the blank back in the spectrometer. Click the begin data collection button (triangle) in the toolbar labeled "Collect". When the absorbance reading stabilizes, click the KEEP button located in the toolbar just to the right of the Red Stop Button (be careful that you **don't** accidentally click the stop data collection button (square)). Type in the concentration of the solution that is in the cuvette (don't include units). OK.

        Pour your samples back into the appropriate TT after you have measured each absorbance and discard them in the waste container only after you have acceptable data.

c. Working in order of most dilute to most concentrated of the solutions, rinse and then fill a cuvette with the solution that will be analyzed. Place the cuvette in the spectrometer. When the absorbance reading stabilizes, click the KEEP button (be careful that you **don't** accidentally click the stop data collection button (square)), and enter the concentration of the analyzed solution. Repeat until the absorbance of each solution has been determined.

d. When the absorbance of all four solutions has been measured, click the stop data collection button (square) located in the toolbar.

e. To determine the equation of the line for your calibration curve click: Analyze ; Linear Fit. A box should appear with the equation and a correlation.

To receive full credit for this lab your calibration curve must be a good, straight-line graph, with a correlation coefficient of 99% or better (Corr: on the screen reads 0.9900 or greater). You should repeat the experiment until you get this proficiency. Work carefully. If you need to repeat the experiment, you still must be done with the write up and post-lab questions before the end of the lab period.

9. Each lab partner's report must have a Logger Pro generated printout of the calibration curve attached to it. The printout must show the graph, the information needed to generate the equation (slope and intercept) for the line, and the correlation reading. To do this click: File ; Print. Uncheck the "Print Visible Spectrum on Wavelength Graphs" and change the "orientation" to landscape under properties. Be sure that the names of all lab partners are entered in the "Name" section and that the date box is checked.

10. When you are done, return your bin to Lab Services.

DATA AND ANALYSIS SHEET: MEASUREMENT (Part II)

Name: _____

Date _____ Lab Partner _____

VOLUME

Balance #_____ Room Temperature_____

Measuring Devise	Mass of 10.0 mL DI water			Average Mass
	1	2	3	
Beaker				
Cylinder				
Pipet (Mohr)				
Pipet (volumetric)				
Buret				

DENSITY

Deionized water (DI water):

Which measuring device most accurately dispenses 10.0 mL of DI water? Why?

Volume of DI water dispensed: _____

Mass (average) of 10.0 mL of DI water using this measuring device: _____

Calculated density of DI water: _____

Density of DI water by hydrometer: _____

Salt Solution:

Identity of salt solution: _____

Mass of 10.0 mL of pipeted salt solution: _____

Calculated density of salt solution: _____

Density of salt solution using a hydrometer: _____

CALIBRATION CURVE

Concentration of stock dye solution: **100%**

Print out and attach the sheet of assigned concentrations (printed in the lab from the lab page of the web site) of dilute dye solutions. Each student must have a copy of the assigned concentrations attached.

Assigned concentrations of dilute dye solutions: _____ , _____ , _____ , _____

Calculate the volume of the stock solution needed to prepare 10.0 mL of each assigned solution (**show your work below**):

Wavelength of maximum absorbance: _____

Equation of line for the calibration curve: _____

Correlation: _____

Attach the calibration curve. The printout must show the graph, the information needed to generate the equation for the line, and the correlation reading. Be sure that the graph title and the names of all lab partners are entered in the footnote and that the date box is checked.