Experiment 7: Solution Preparation

Purpose

Scientists in many different fields use solutions for their laboratory work. Solutions have a solute dissolved in a solvent, and the mixture is homogeneous. For our general chemistry laboratory experiments, the solutions are aqueous, meaning that the solute is dissolved in water.

This experiment is an exercise in preparing solutions, starting with the preparation of a stock solution. Diluted solutions will then be prepared with the stock solution. These diluted solutions will let you practice solution preparation. A spectrophotometer will be used to measure the amount of light absorbed by your solutions. This will let you "see" how well your solutions were prepared. You will also need to prepare standard solutions (solutions of known concentration) to make a calibration curve that lets you determine the concentration of a red dye solution of unknown concentration.

Background

Solution preparation will involve the dilution of the concentrated stock solution. The dilution equation will be used to determine the concentration of the new, diluted solution.

$(M_c)(V_c) = (M_d)(V_d)$ where M_c is the concentration of the concentrated stock solution, V_c is the pipeted volume of the stock solution, M_d is the concentration of the new diluted solution, and V_d is the volume of the new diluted solution. The concentration unit does not have to be molarity. However, M_c and M_d must be the same concentration unit. V_c and V_d do not have to be in the unit of liters, but they both have to be the same volume unit.

The absorbance of light will follow the Beer-Lambert Law (Beer's Law), which states that the absorbance of light by a solute in solution is directly proportional to its concentration. The mathematical equation is $A = abc$, where A is absorbance, a is the molar absorptivity coefficient, b is the path length, and c is concentration.

Calibration curves are graphs that show the relationship between concentration and absorbance of light by the solute. Absorbance is dependent on the concentration of solute in the solution. Therefore, concentration is on the x-axis and absorbance is on the y-axis. The data points obtained in lab are plotted using Excel or Google Sheets. The computer program will put a best-fit line through the data points. The equation of the linear line is obtained, and this equation shows the mathematical relationship in the

$y = mx + b$ format which translates to $A = mc + b$

The spectrophotometers that are in the general chemistry laboratory cover the wavelength range of visible light. Therefore, in order to measure the absorbance of light by the solute in solution, the solute must absorb visible light.

The **first step** to prepare a calibration curve is to make the standard solutions. These are solutions of known concentration. The **second step** is to determine the best wavelength at which to measure the absorbance. The best wavelength is typically the

wavelength at which the solute absorbs the maximum amount of light, which is referred to as λ_{max}. The **third step** measures the absorbance of light by the standard solutions and sample solutions, using the spectrophotometer set to λ_{max}.

Chemicals

Red food dye #1 (liquid form) Deionized water

Equipment

Pipets and pipettors	Volumetric flasks, 100 mL
Parafilm	Plastic droppers
Beakers, small sizes	Laboratory balance

Procedure

Preparation of the stock solution

1) Bring your laboratory notebook, pen, plastic dropper, and a clean, dry, 150 mL beaker to the laboratory balance.
2) Place the beaker on the balance pan, and tare the balance so the beaker has a mass of zero grams.
3) Use your clean plastic dropper to put 0.50 g of liquid red dye into your beaker. (This will be approximately 12 drops.) Record the exact mass of the red dye in your notebook. Do not tare the balance again.
4) Remove the beaker from the balance pan, and add approximately 40 mL of deionized water using a graduated cylinder.
5) Return the beaker to the balance pan, and gently add deionized water to the beaker until the total mass in the beaker is 50 g. The mass does not have to be exactly 50 grams, but the exact total mass must be recorded in your notebook. (Use all digits displayed on the balance.)
6) Return to your lab bench. Use a clean, dry, glass stir rod to mix your stock solution well.
7) Calculate the concentration of the stock solution, in units of % wt.

Serial Dilutions (Save all of these solutions; you will need them throughout the experiment.)

1) Use a calibrated pipet to measure and dispense your stock solution for this part of the experiment (*do not use a plastic dropper*). Rinse the pipet with your stock solution, and then dispense it to a waste beaker.

2) Prepare serial dilution (1). Draw up your stock solution into the pipet and adjust the bottom of the meniscus to be exactly on the 5.0 mL mark. Dispense this volume to a 100 mL volumetric flask. (The flask should already have been rinsed with DI water. The flask does not have to be dry, since you will be adding water to it.) Dilute the contents of the flask with DI water until the bottom of the meniscus sits exactly on the calibration mark on the next of the flask. The total

volume of solution in the flask will now be 100.0 mL. Cover and seal the flask with Parafilm. Invert to mix fifty times.
Calculate the concentration of serial dilution (1). Use the dilution equation, Mc·Vc=Md·Vd. *(The %wt unit works fine for Mc and Md.)*

3) Prepare serial dilution (2). Rinse the pipet with DI water, and then rinse with your serial dilution (1) solution. Draw up your (1) solution into the pipet and adjust the bottom of the meniscus to be exactly on the 5.0 mL mark. Dispense this volume to a 100 mL volumetric flask. Dilute the contents of the flask with DI water until the bottom of the meniscus sits exactly on the calibration mark on the next of the flask. The total volume of solution in the flask will now be 100.0 mL. Cover and seal the flask with Parafilm. Invert to mix fifty times.
Calculate the concentration of serial dilution (2). Use the dilution equation, Mc·Vc=Md·Vd.

4) Prepare serial dilution (3). Rinse the pipet with DI water, and then rinse with your serial dilution (2) solution. Draw up your (2) solution into the pipet and adjust the bottom of the meniscus to be exactly on the 5.0 mL mark. Dispense this volume to a 100 mL volumetric flask. Dilute the contents of the flask with DI water until the bottom of the meniscus sits exactly on the one calibration mark on the next of the flask. The total volume of solution in the flask will now be 100.0 mL. Cover and seal the flask with Parafilm. Invert to mix fifty times.
Calculate the concentration of serial dilution (3). Use the dilution equation, Mc·Vc=Md·Vd.

Determining the λ_{max} (finding the best wavelength for absorbance of light by red dye)

1) Use the Serial Dilution (1) solution for this part of the experiment. Place some of this solution into a small test tube, about ¾ full. Put DI water into another test tube, also about ¾ full. The DI water is your blank that will be used to zero the spectrophotometer.
2) Red dye looks red because it absorbs light in the blue-green region of visible light (see Figure 1). You need to determine the best absorbance. Turn on the spectrophotometer and set the wavelength to 440 nm.

Figure 1

Coloured absorbed	Visible (Complementary)
Violet	Yellow-Green
Blue	Yellow
Green-Blue	Orange
Blue-Green	Red
Green	Purple
Yellow-green	Violet
Yellow	Blue
Orange	Green-Blue
Red	Blue=Green

https://www.slideshare.net/GranchBerheTseghai/2-fundamentals-of-color

3) Put the test tube with the DI water into the sample compartment of the spectrophotometer, and close the lid. Press the zero Abs button. This sets the zero point on the spectrophotometer, which subtracts out any absorbance of light by the glass test tube and DI water.
4) Put the test tube of red dye solution into the sample compartment and close the lid. Record the absorbance readout in your notebook.
5) Repeat steps 8 and 9 after increasing the nm setting by 10 nm. Continue to do this until you have absorbance readings up to 560 nm. You must set the blank each time the nm setting is changed. Fine tune the maximum absorbance wavelength once you think you have found λ_{max}.
6) Plot the absorbance vs. nm on graph paper. Report the best wavelength to use when measuring the absorbance of light by the red dye solution.

Preparing Standard Solutions and a Calibration Curve

Solutions of known concentration will be prepared to make a calibration curve. Zero the spectrophotometer with the DI water blank.

Use the stock solution to prepare the standard solutions.

1) Standard Solution 1: Pipet 1 mL of stock solution into a 100 mL volumetric flask and dilute to mark with DI water. Seal the flask and invert 50 times to mix well.
2) Standard Solution 2: Pipet 2 mL of stock solution into a 100 mL volumetric flask and dilute to mark with DI water. Seal the flask and invert 50 times to mix well.
3) Standard Solution 3: Pipet 3 mL of stock solution into a 100 mL volumetric flask and dilute to mark with DI water. Seal the flask and invert 50 times to mix well.
4) Standard Solution 4: Pipet 4 mL of stock solution into a 100 mL volumetric flask and dilute to mark with DI water. Seal the flask and invert 50 times to mix well.

Measure the absorbance of light for each standard solution and the unknown solution.

1) Make sure the spectrophotometer is set to your λ_{max} for the wavelength.
2) Use the DI water as the blank to zero the spectrophotometer.
3) Transfer each standard solution and unknown into its own test tube, about ¾ full.
4) Measure the absorbance of light for each solution, and record these values into your notebook.

Prepare a calibration curve.

Follow the instructions in the Calibration Curve video and use Excel or Google Sheets to prepare a calibration curve. The concentration is the independent variable, so that is your X axis. Absorbance is the dependent value, so that is the Y axis. Plot a scatter plot, linear, and put the best-fit line on the graph. Get the equation of the line and the R^2 value.

Determine the concentration of your unknown.

Use the equation of the line to solve for the unknown concentration. The equation of the line is in the y=mx+b format. Y is the absorbance and X is the concentration. You know the absorbance of the unknown solution, so solve for

X. Show this calculation in your notebook, and report the concentration of the unknown with correct significant figures and units.

Determining the Proper Mixing Amount for a Volumetric Flask

Convention states that a person is to seal the volumetric flask and invert, to mix the contents in the neck and bulb parts of the flask. This back and forth invert / upright mixing is supposed to be done 50 times to ensure a homogeneous mixture. Does this have to really be done 50 times? That's what this part of the experiment will determine.

1) Use your stock solution for this part. Pipet 5.0 mL of stock solution into a 100 mL volumetric flask. Dilute to volume with DI water. DO NOT seal with Parafilm, DO NOT mix.
2) Use a plastic dropper inserted into the neck of the flask to take out enough solution to fill a small test tube ¾ full. (test tube A)
3) Use the same plastic dropper to remove enough solution to fill a second small test tube ¾ full. (test tube B).
4) Use the same plastic dropper to remove enough solution to fill a third small test tube ¾ full. (test tube C) *It may be necessary to tilt the volumetric flask so the dropper can reach the solution inside the flask.*
5) Repeat steps 1-4 to prepare another solution. However, for this solution, seal the volumetric flask with Parafilm, invert to mix 30 times. Then remove the solution into three different test tubes (test tubes D, E, F)
6) Repeat steps 1-4 to prepare another solution. However, for this solution, seal the volumetric flask with Parafilm, invert to mix 50 times. Then remove the solution into three different test tubes. (test tubes G, H, I)
7) Set the spectrophotometer to the λ_{max} that you determined previously.
8) Measure the absorbance for each test tube, A – I. Record these absorbances in your notebook.
   For each set of three solutions, are the absorbance readings the same?

Notes:

1) For your Results and Calculations section of your notebook, show all concentration calculations for the different solutions you prepared. Also, be sure to show the calculations for determining the unknown's concentration.
2) For your Conclusion section of your notebook, discuss the conclusions made for each section of this experiment. Such as, what is the λ_{max} that you determined and how do you know? What is a good concentration range to use for red dye in the spectrophotometer, and how do you know (serial dilution section)? How many times do you really need to invert a volumetric flask to properly mix a solution, and how do you know? How good was your calibration curve (check R^2, does R^2 equal 1.000)? Does your calculated unknown concentration make sense when you compare the color of the unknown solution to the colors of your standard solutions?