Chem 112 – Experiment 2 – Simulation – Spectrophotometric Analysis of Copper

Background

General

An experiment that I always wanted to introduce into Chem 112 was "Synthesis of a Copper Coordination Complex" however the subsequent analysis of the copper in the complex involved Spectrophotometric Analysis, involving the use of a UV Spectrometry that the sheer size of the Chem 112 class and the cost of these spectrometers precluded. Instead it was an experiment that was confined to Chem 121H.

This experiment involves some background theory that you may not meet in class and thus to facilitate understanding this process I would like to introduce you to a former college of mine, Prof. William Vining who has developed a beautiful introduction to this with some novel experiments and even a section that shows you how you could potentially use a smart phone to make crude spectrophotometric analysis. This is just background material for you and is not part of the laboratory report.

https://iw9zkf.axshare.com/introduction.html
If asked for a password use: sunnyday

I am grateful to Prof. Vining for his permission to use this.

Spectroscopy

A solution appears colored because it absorbs certain wavelengths of light in the visible spectrum while transmitting or reflecting others. The absorbance or transmission of light at specific wavelengths is measured using spectrophotometry. Spectrophotometry data can be used to determine the concentration of a colored substance in solution.

Spectrophotometry is a technique that measures the amount of light absorbed by a colored sample. The more concentrated a colored substance in a solution is, the more light it absorbs. Therefore, this technique can be used to analyze sample solutions of unknown concentration. The proportion of light that passes through a solution is called transmittance, whereas the proportion of light that is absorbed is called absorbance. A spectrophotometer is an instrument that quantitatively measures the proportion of light that passes through a solution at different wavelengths. In a spectrophotometry experiment, the sample solution is contained in a cuvette which is placed in the spectrophotometer. Light from a lamp is focused on the sample, and the light transmitted through the sample is detected.

Transmittance is often expressed as a percentage and is calculated as shown below.

$$\%T = (I/I_0) \times 100$$

where

T : is transmittance.
I_0 : is intensity of light received by solution.
I : is the amount of light transmitted by the substance.

Absorbance is related to **transmittance** by **the equation below**.

$$A = 2 - \log_{10} T$$

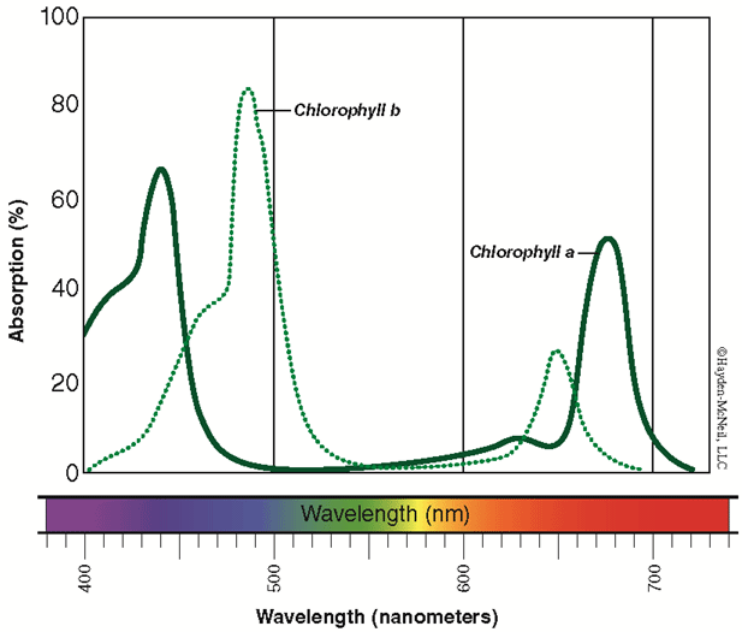
where

A : is absorbance.

Based on the equation above, A has values between 0 and 2.
Using a spectrophotometer, both %T and A can be measured.

Absorption Spectrum

The plot of the absorbance of a solution at a range of wavelengths is called an absorption spectrum. A colored solution will have one or more absorption maximum (λ_{max} or lambda max) in the visible spectrum. Visible wavelengths cover a range from approximately 400 to 700 nm. The longest visible wavelength is red and the shortest is violet. The figure below illustrates this range as well as the absorption spectra of chlorophyll a and chlorophyll b.



Beer's Law

Absorbance is related to concentration as defined by Beer's law, shown below.

$$A = \epsilon c l$$

Where

A: is the measured absorbance.
ε: is the molar extinction coefficient in $M^{-1}cm^{-1}$.
c: is the molar concentration in mol/L.
l: is the path length in cm.

The molar extinction coefficient and path length are constant for a given substance and experimental setup. Therefore, if the absorbance of a standard solution with a known concentration is measured, an unknown concentration can be determined using the equation below.

$$\frac{A_1}{A_2} = \frac{c_1}{c_2}$$

Where

A1 and c1: are the absorbance and concentration of the known concentration solution.
A2 and c2: are the absorbance and concentration of the unknown concentration solution.

In addition, a set of standard solutions of known concentrations can be used to create a standard curve of absorbance versus concentration. Given that Beer's law states that absorbance is directly proportional to concentration, this standard curve produces a straight line with a slope equal to $\varepsilon \times l$.

About This Lab

In this lab, you will first determine the wavelength of maximum absorbance of two solutions. Next, you will measure the absorbance of copper solutions of known concentrations and prepare a calibration curve. You will then use this data to determine the concentration of copper ions in the two samples of unknown concentration from their absorbance measurements.

Open the simulation by clicking on the virtual lab icon shown on the left on the Hayden-McNeil Web Site. The simulation will launch in a new window.

You may need to move or resize the window in order to view both the Procedure and the simulation at the same time.

Follow the instructions in the Procedure to complete each part of the simulation. When instructed to record your observations, record data, or complete calculations, record them for your own records in order to use them later to complete the post-lab assignment.

Procedure

Experiment 2_1 – Determine the λ_{max} of Colored Solutions.

1. Take **two cuvettes** from the **Containers shelf** and place them on the workbench.

2. Take **0.06 M copper(II) sulfate** from the **Materials shelf** and **add 3 mL to the first cuvette**.

3. Take **0.025 M cobalt(II) chloride** from the **Materials shelf** and add **3 mL to the second cuvette**.

4. Take a **spectrophotometer** from the **Instruments shelf** and place it on the workbench.

5. Place a **third cuvette** from the **Containers shelf** onto the workbench. **Add 3 mL water** to this cuvette. ***This is the blank cuvette***.

6. Insert the **blank cuvette** in the **spectrophotometer**. Make sure the **spectrophotometer output** is **set to absorbance** (A) and **set the wavelength to 400 nm** either by moving the slider or by clicking on the wavelength value and typing the new value. **Press the Zero button**. Remove the cuvette and place it on the workbench.

7. Collect absorbance versus wavelength data for the copper(II) sulfate solution as follows:
   a. Move the **cuvette with the 0.06 M copper(II)** sulfate solution into the **spectrophotometer**.
   b. **Record the absorbance** for the solution.
   c. **Increase the wavelength to 420 nm**. **Record the absorbance and corresponding wavelength**.
   d. **Continue increasing the wavelength in 20 nm increments** and **recording the absorbance** at each setting **until you reach 700 nm**.
   e. Remove the cuvette from the spectrophotometer and place it on the workbench.

8. Move the cuvette with the **0.06 M cobalt(II) chloride solution into the spectrophotometer** and **repeat the process in step 7** to measure the absorbance at each 20 nm increment. Remove the cuvette from the spectrophotometer and place it on the workbench.

9. Clear the cuvettes from the bench by emptying them into the waste container and then dragging them to the sink.

10. Review the absorbance data you collected for both copper(II) and cobalt(II) ions, and **determine the wavelength at which the absorbance reading was the highest**. **This is your λ_{max}** (lambda max). **Record the λ_{max} value for both copper and cobalt ions**.
    Note: If a well-defined peak does not exist it is acceptable to use the wavelength that corresponds to the highest measurable absorbance value.

Experiment 2_2 – Measure Absorbance versus Concentration for Cu^{2+} Ions.

1. Take **three** clean **50 mL volumetric flasks** from the **Containers shelf** and place them on workbench.

2. Fill the flasks with the following amounts of 0.06 M copper(II) sulfate and 1 M nitric acid:

	$CuSO_4$ (mL)	HNO_3 (mL)	Cu^{2+} (M)
Flask 1	20	20	?
Flask 2	15	5	?
Flask 3	10	0	**0.06**

3. Obtain **3 cuvettes** from the **Containers shelf**. **Transfer 3 mL** from the **first flask** into the **first cuvette**, from the **second flask** into the **second cuvette**, and from the **third flask** into the **third cuvette**.

4. Use the dilution formula, $C_1V_1 = C_2V_2$, to **calculate the concentration of Cu^{2+}** ions in **cuvettes 1 and 2**. **Record** these **concentrations**.
   Use the table to record these concentrations.

5. Obtain **another cuvette** from the **Containers shelf** and place it on the workbench. **Add 3 mL water**. **This is the blank cuvette**.

6. **Place** the **blank cuvette** into the **spectrophotometer**. *Check to make sure the spectrophotometer output is still absorbance (A)*. **Set the wavelength to λ_{max} for copper ions that you determined in the previous experiment. Press the Zero button**.

7. **Remove the cuvette** from the spectrophotometer and place it on the workbench. **Insert the other three cuvettes in sequence**, **recording the absorbance each time**.
   Be careful also to note which concentration of Cu^{2+} ions is in each cuvette.

8. Clear the cuvettes from the bench by emptying them in the waste then placing them in the sink.

Experiment 2_3 – Determine the Cu^{2+} Concentration in Unknown Solutions of Copper(II)Sulfate.

1. Take **three clean cuvettes** from the **Containers shelf** and place them on the workbench.

2. Take **copper solution #1** from the **Materials shelf** and **add 3 mL to the first cuvette**.

3. Take **copper solution #2** from the **Materials shelf** and **add 3 mL to the second cuvette**.

4. **Add 3 mL water** to the **third cuvette** as a **blank**.

5. **Insert the blank cuvette** into the **spectrophotometer**.
   Make sure that the spectrophotometer is set to the wavelength corresponding to λ_{max} for Cu^{2+} ions.

6. **Press** the **Zero button**. Remove this cuvette.

7. Insert the next cuvette into the spectrophotometer and **record the absorbance** of the unknown solution. Repeat with the final cuvette.

8. Clear the bench of all materials, containers, and instruments, then go to the **General Chemistry web site** and **download the Data file**, this when completed is the **report that you send to your TA**.