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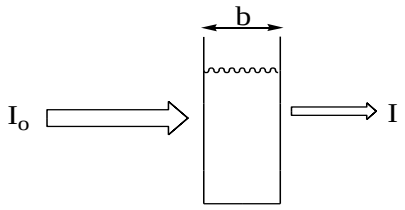
Experiment #6 – Colorimetric Determination of Co^{+2}

Although volumetric and gravimetric mass analyses are commonly used, spectroscopy is the technique most often used for modern chemical analysis. Spectroscopy is the study of electromagnetic radiation emitted or absorbed by a given chemical species. Since the quantity of radiation absorbed or emitted can be related to the quantity of the absorbing or emitting species present, this technique can be used for quantitative analysis. There are many spectroscopic techniques available from X-rays, ultraviolet, infrared, and visible light, to name a few. We will consider one form here which is based on the absorption around visible light.

If a liquid is colored, it is because some component of the liquid absorbs visible light. In a solution, the greater the concentration of the light-absorbing substance, the more light absorbed and the more intense the color of the solution. The quantity of light absorbed by a substance can be measured using a spectrophotometer. The instrument consists of: (1) a source that emits wavelengths of light in a measurable region (i.e. visible light which has wavelengths 400 to 700 nm); (2) a monochromator which selects a given wavelength of light; (3) a sample holder for the solution being measured; and (4) a detector which compares the intensity of incident light I_o to the intensity of light after it has passed through the sample I. When a beam of light passes through a substance, some of the energy is often absorbed by the substance. This causes a decrease in the intensity of the transmitted beam. The ratio I / I_o is called the transmittance, T, a measure of the fraction of light that passes through the sample holder (or cuvette) which contains the absorbing solution. The amount of light absorbed by the solution is given by the absorbance, A, where:

$$A = -\log (I / I_o) = -\log T \qquad (1)$$



Absorbing Solution
of concentration c

$T = I/I_o$

$A = -\log (I/I_o) = -\log T$

$\%T = 100\% T$

The distance, b, the light travels through the solution (in cm) and the concentration, c, of the absorbing species (in mol / L) are represented in the schematic above. A beam of parallel radiation with an intensity is shown before (I_o) and after (I) it has passed through a layer of solution with a measured thickness at a certain concentration. The Beer-Lambert law is the basis for using spectroscopy in quantitative analysis which relates absorbance (A) to the concentration of the absorbing solution (c) and the path length of the cuvette (b). That is:

$$A = \varepsilon b c \quad (2)$$

where ε is the molar absorptivity or the molar extinction coefficient (in L / mol·cm). Each pure substance has its own unique extinction coefficient. Note that during the experiment, the same cuvette should be used for all measurements. With the same cuvette, the path length (b) and the extinction coefficient (ε) remain constant. Therefore, we can mathematically say that ε b = k (constant). If we write the concentration (c) as M for molarity, our new equation becomes:

$$A = k M \quad (3)$$

Once absorption values for different concentrations are obtained, a Beer's law plot of absorbance (vertical axis) versus concentration (horizontal axis) is made. A best-fitting line of the data points is constructed, from which you can determine your equation in slope-intercept form A = (ε b) c + 0 or A = k M + 0. By measuring the path length of your cuvette, the extinction coefficient can then be calculated.

In this experiment, there will be three basic tasks to accomplish using the spectrophotometer. First, students will collectively determine the wavelength at which 0.100 M $Co(NO_3)_2$ will absorb best. Next, a standard absorbance curve from which the extinction coefficient can be calculated will be constructed. Finally, an unknown $Co(NO_3)_2$ solution will be analyzed for concentration determination.

Procedure

Part I. Prepare a standard set of $Co(NO_3)_2$ solutions

Using a buret containing the stock solution of 0.100 M $Co(NO_3)_2$ and a 10.00 mL volumetric flask, prepare the following solutions of Co^{2+} by dilution: 0.0800 M, 0.0600 M, 0.0400 M, 0.0200 M.

Part II. Calibrate the Spectrovis

**** If you are the second user of the Spectrovis, you may skip Part II of this procedure and start at Part III Step 1 using the solutions that you prepared in Part I.***

1. Plug the USB cable from the Spectrovis into the PC.
2. Open Logger Pro on the computer.
3. Under Experiment choose Calibrate then Spectrometer 1.
4. Allow the 90 s warmup.
5. Fill the cuvette (up to about ¾) with the blank solution (blank is just deionized water for this experiment). Insert the cuvette into the Spectrovis. Make sure the clear sides are the ones in the light path.
6. Select Finish Calibration.
7. When OK becomes available, select it.
8. The machine is now calibrated and there is no need to re-calibrate unless you exit out of Logger Pro.

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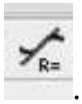
Part III. Determination of Maximum Absorbance Wavelength.

To obtain a ***spectrum and determine λ_{max}***:

1. Discard the blank solution and use the same cuvette for the next steps.
2. Fill your cuvette (up to about ¾) with the stock 0.100 M Co^{2+} solution.
3. Insert the cuvette into the Spectrovis.
4. Select the green button on the computer screen to start collection.
5. Once the spectrum is stabilized (usually takes a second), click stop.
6. To determine the wavelength where absorbance is maximum (λ_{max}), move your cursor over the peak and write down the λ (the reading should be down in the lower left hand corner).

Part IV. Standard Calibration Curve

1. Click the Configure Spectrometer button  .
2. Choose the Absorbance vs Concentration option.
3. ON the bottom drop down menu, change from single 10 nm into individual wavelengths.
4. Choose the λ_{max} you determined from the previous step.
5. Click OK.
6. If one or two pop-up window/s appear/s, choose Erase and Continue AND/OR "No" on the next pop-up window.
7. Click collect (green button).
8. Fill (up to about ¾) your cuvette with the stock (0.100 M) Co^{2+} solution and insert the cuvette into the Spectrovis.
9. When the absorbance reading stabilizes (usually takes a second), click KEEP.
10. On the pop-up window, enter the concentration of the sample and click OK. ***DO NOT CLICK STOP.*** You will click STOP in step 13.
11. Discard the solution in the cuvette.
12. Repeat steps 8 through 11 for each of the standard solutions that you prepared. Also, repeat step 8 through 11 for deionized water (a 0.000 M solution of Co^{2+}).
13. When you have measured ***ALL*** of these solutions, Click STOP to end data collection.
14. Click Linear Fit  . If a pop-up window appears, choose the "latest run" option.
15. Write down the slope of the line (what is the slope equal to?).

Part V. Determination of Unknown [Co^{2+}]

1. Fill the cuvette with your unknown solution.
2. Insert the cuvette into the Spectrovis.
3. Click collect. If a pop-up window appears, choose "store latest run."
4. Write down the absorbance (shown on the bottom left corner of the screen).
5. Calculate the concentration of your unknown solution using Beer's Law.

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Data and Calculations

Wavelength of maximum absorbance is _____.

$[Co^{+2}]$	Volume of 0.100 M $Co(NO_3)_2$ Stock Solution	ABSORBANCE
0.1000 M	_____ mL	_____
0.0800 M	_____ mL	_____
0.0600 M	_____ mL	_____
0.0400 M	_____ mL	_____
0.0200 M	_____ mL	_____
0.0000 M	_____ mL	_____

UNKNOWN # _____ ABSORBANCE _____

Path length of the cuvette: _____

Using Microsoft Excel, plot a graph of absorbance (y) verses concentration (x). Using the graph plotted from your data and the path length of the cuvette, calculate the extinction coefficient.

Extinction Coefficient: _____

Solve for the concentration on your unknown solution…

(a) $[Co^{+2}]$ _____(read from graph)

(b) $[Co^{+2}]$ _____ (calculate from line equation and slope value)

SHOW CALCULATIONS:

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Post-Lab Questions: Colorimetric of Co^{+2}

1. Calculate the transmittance of a solution if its absorbance is 0.352.

2. Calculate the absorbance of a solution if the transmittance is 0.647.

3. The following absorbance values for four solutions with known MnO_4^- concentrations were measured using a spectrophotometer:

Solution	[MnO_4^-]	Absorbance
1	0.700×10^{-4} M	0.175
2	1.00×10^{-4} M	0.250
3	2.00×10^{-4} M	0.500
4	3.50×10^{-4} M	0.875

   A. Using Microsoft Excel, plot a graph of Absorbance vs. Concentration of MnO_4^-. Write the trendline linear equation from the plotted graph.

   B. Determine the slope of the graph and include its units.

   C. Determine the concentration of an unknown MnO_4^- sample whose absorbance is 0.780.

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D. Using the graph paper, below, construct a graph of Absorbance vs Concentration of MnO_4^-. Draw a linear trendline and determine the equation of the line that you drew. How does this compare to the graph that you made using Excel?