

Experiment 9

Spectrophotometric Determination of MW of Cobalt Complex

Adapted from *Manual for Laboratory Investigations in General Chemistry*, Shakhshiri, B.Z. and Dirreen, G. E., 1982.

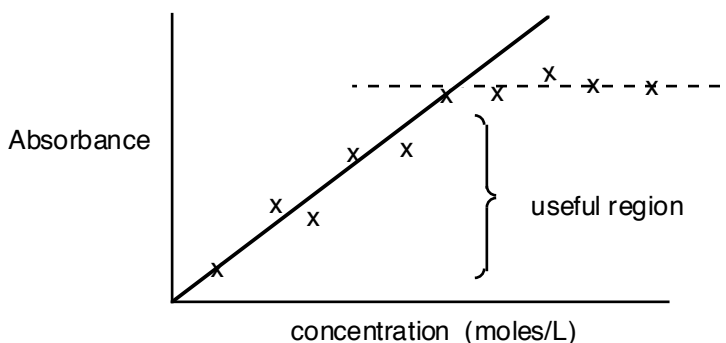
Discussion

Many metal coordination complexes absorb energy in the visible part of the electromagnetic spectrum, and therefore appear colored. We can make use of this characteristic in an analytical method known as spectrophotometry. The relationship between absorbance and concentration of an absorbing species is given by Beer's Law:

$$\text{Absorbance (A)} = \epsilon l c$$

where ϵ = the molar absorptivity (in units of $M^{-1}cm^{-1}$), l = pathlength (in cm), and c = concentration of the solution (in M). The absorbance is determined by measuring the light absorption at a fixed wavelength for a blank solution (solvent alone) and for a solution of the complex. The difference in absorbance between these two solutions is the absorbance for the complex alone. If ϵ is known for a given complex, then the concentration of a given solution may be determined (because l is fixed for a sample cell – usually $l = 1cm$).

In order to find ϵ for your complex, you will prepare a standard curve. You will be given stock solution of compound X and Y, from which you will prepare a series of solutions of known concentration. You will take absorbance readings on these solutions, and prepare a plot of Absorbance vs. concentration. This plot is known as a standard curve, and should look something like the figure shown below. From this plot, you can determine the slope of the solid line, which is simply ϵ . Note that at higher concentrations, the absorbance readings begin to flatten out; the standard curve should not be used in this region.



Once you have prepared your standard curve, you will prepare a solution of a sample of your cobalt complex of known mass in a known volume. You can then measure the absorbance of your solution, and using your standard curve, determine the concentration of your solution. Once you have the concentration (moles/L) of your solution, you can use this information together with the mass of cobalt complex you started with, and the volume of your solution in order to determine the MW of your complex as indicated below:

$$\begin{array}{cccc} \text{mass of original} & \times & (\text{volume of solution})^{-1} & \times & (\text{concentration})^{-1} & = & \text{molecular weight} \\ \text{cobalt complex} & & & & & & \\ \text{(grams)} & & \text{(1/L)} & & \text{(L/moles)} & & \text{(grams/mole)} \end{array}$$

Procedure

Standard Curve

You will be provided with a 10 mM stock solution of compound X and Y. Prepare 10 mL of 1,2,5,8, and 10 mM solutions from this stock solution using the volumetric glassware provided. Use these solutions to generate your standard curve; use distilled water as your "blank" solution. Measure a spectrum of your solution to pick an appropriate wavelength for your analysis. Generate a plot of the absorbance at your chosen wavelength as a function of concentration.

Analysis of cobalt complex

Accurately weigh out approximately 20-30 mg of your cobalt complex (X or Y) onto weighing paper and transfer the sample into a volumetric flask. Fill the flask about half way with water and heat gently until the solid has completely dissolved. Fill the volumetric flask to the line with distilled water, and mix well. You will have to cool the solution to room temperature for an accurate volume measurement. Run a blank (distilled water), then run a UV-Vis spectrum of your solution. Your data must be within the linear portion of your standard curve in order to do the analysis. If your solution is off-scale compared to your standard curve, you should do a 10-fold serial dilution of your solution, and run the spectrum again.

Experiment 9 Worksheet — Spectrophotometric Analysis

Name _____

Date of Experiment: ___/___/___

Report: ___/___/___

Wavelength chosen for analysis: λ_{\max} = _____

Data for standard curve

Concentration of Complex

Absorbance at λ_{\max}

value for molar absorptivity at λ_{\max} _____
(attach graph of standard curve)

Data for unknown Cobalt Complex

Absorbance at λ_{\max} _____

Concentration (from standard curve) _____

MW of cobalt complex _____
(show calculations below)

Identity of compound based on spectrophotometric analysis _____

