Introduction to Spectroscopy

Introduction

The wavelength of light that an atom absorbs or emits is related to the distribution and movement of electrons within that atom. Thus, we can explain the observed colors of objects using submicroscopic concepts from chemistry and physics. In this experiment, you'll enjoy learning about visible light spectroscopy, a field of science founded on the interaction between light and the electrons in atoms, molecules, and ions. Visible light spectroscopy is a common analysis method, with many industrial, medical, biochemical, and chemical applications.

Although we cannot see electrons moving around nuclei, we effectively extend our senses using a visible light spectrophotometer. Learning to operate this new, high-tech piece of equipment--the Ocean Optics spectrometer and its associated software--has the potential to pay real dividends. As you work through the five parts of this experiment, you will see how spectroscopy can be applied to determine the chemical identities of atoms, ions, and molecules (Part 1). Spectroscopy is also useful for determining concentration (Parts 2 and 3), and can be used in studies of the chemical and physical behaviors of substances (Part 4). Because we are studying visible light spectroscopy, you will work with colored solutions in this experiment.

Pre-lab

Safety

Goggles must be worn at all times. Please do not uncap or discard the solutions in the pre-filled cuvettes. The other solutions that you work with in this lab may be rinsed down the drain.

1. List all of the chemicals you will use for this week's experiment. For each chemical, list specific safety precaution(s) that must be followed. In order to find specific safety information, please obtain a Materials Safety Data Sheet (MSDS) on the chemical of interest. MSDSs can be found through an internet search (e.g., google) or from the following website: www.hazard.com Read the MSDS and find specific safety concerns for each chemical.

2. Click here for information on how the visible spectrum relates to other forms of electromagnetic radiation. Perform an internet search on “visible light spectrum” using Google Images or another search engine. Become familiar with the colors of the visible spectrum, their order, and their wavelengths.

3. Using your favorite web search engine, search for sites containing the words spectrophotometry, spectroscopy, spectrophotometer, or spectrometer; if you wish, you may include visible, ultraviolet, or UV as adjectives in your search term. (At this point, it is not necessary to explore spectroscopy involving mass (MS), nuclear magnetic resonance (NMR), infrared (IR), or Raman spectroscopy.) For this week's prelab, record the URLs of at least two websites that you found interesting, and very briefly describe what you learned at each site.

4. Read the instructions for the operation of the Ocean Optics Spectrophotometer.
Procedure

Spectrophotometers like the Ocean Optics systems are capable of "seeing" the same colors that the human eye sees. Completing Part 1 will help you and your lab partners recognize how information from the spectrophotometer can be correlated with the observed color of aqueous solutions.

Open the LoggerPro software and prepare the Ocean Optics spectrophotometer (Click here for instructions.) Prepare a spectrometer calibration blank simply by filling a rinsed cuvette approximately two-thirds full with distilled water. Calibrate the spectrophotometer; for help, see question #4 of this week’s Pre-Lab.

Part 1 – Assessing the Impact of Color in Spectrophotometry

Obtain a pre-filled cuvette of each of the following solutions: 0.10 M Co(NO$_3$)$_2$, Cu(NO$_3$)$_2$, Ni(NO$_3$)$_2$, and K$_2$CrO$_4$. (Please do not uncap or discard the solutions in the pre-filled cuvettes.) Record the color of each solution. Now place one of the colored solution cuvettes into the spectrometer and view its transmittance spectrum (viewable by clicking Experiment → Change Units → Spectrophotometer → %Transmittance). Determine the wavelength range for which transmittance is greatest. Repeat this process for the other three colored solutions. Talk with your group members about the wavelength ranges for the colors of the visible spectrum. Does the color of a given solution relate to the range of wavelengths transmitted by that solution?

Now view the absorbance spectrum (by clicking Experiment → Change Units → Spectrophotometer → Absorbance) for each of the four colored solutions listed above. At what wavelength does the absorbance exhibit a maximum? What is the relationship between the color transmitted and the color absorbed for each solution? (For help, you may wish to refer to a color wheel like the one shown below.)

![Color Wheel](image)

Note: If you copy and paste any spectra into a Word document to refer to them later, don't forget to delete these spectra files from the computer before you leave!

Part 2 – Diluting a Colored Solution

In this Part, your group will prepare several dilute solutions to investigate the effect of concentration on absorbance. Obtain some 0.10 M Cu(NO$_3$)$_2$ solution from the stock bottle. Now work with your group members to develop a scheme for combining measured volumes of 0.10 M Cu(NO$_3$)$_2$ solution with distilled water to generate four additional solutions: 0.050 M, 0.025 M, 0.015 M, and 0.010 M Cu(NO$_3$)$_2$. As your group decides how these solutions should be prepared, keep in mind that you will need enough of each solution to fill a cuvette about two-thirds full. In addition to filling cuvettes with each of the four dilute solutions, fill one cuvette with the undiluted, 0.10 M stock solution. Before you analyze this series of solutions with the spectrophotometer, note any systematic difference in the appearance of the five Cu(NO$_3$)$_2$ solutions.
Place each cuvette into the spectrophotometer and note the absorbance reading at the wavelength of maximum absorbance for Cu(NO₃)₂--this wavelength should already be recorded in your notes from Part 1. Use Excel to generate a graph of absorbance vs. Cu(NO₃)₂ concentration. Does your plot show a trend? Use mathematics to help you answer this question by adding a trendline to your Excel data.

Part 3 - Analyzing a Cu(NO₃)₂ Solution of Unknown Concentration

Could your data from Part 2 be used to determine the concentration of an unknown solution? Often, quantitative analytical studies employ absorbance vs. concentration plots similar the one your group generated in Part 2. Such a plot is known as a calibration curve. The solutions of known concentration used to acquire the data for the curve are referred to as standards. To use the curve, measure the absorbance of a sample of unknown concentration and then, using graphical interpolation, determine its concentration. (Hint: The process of interpolation will be easiest if your group uses Excel to display the equation associated with the trendline.) Employ this approach to determine the concentration of the unknown Cu(NO₃)₂ solution provided to you.

Part 4 - Solution Spectra Before and After Mixing

Fill a cuvette approximately two-thirds full with 1.0 x 10⁻³ M Fe(NO₃)₃ solution. Fill a second cuvette approximately two-thirds full with 5.0 x 10⁻³ M KSCN. Based on their appearance, what do you expect the absorbance spectrum of each of these solutions look like? Try it and find out! Now mix the contents of these two cuvettes in a small container. Is the absorbance spectrum remarkably different for the mixture?