Introduction to Spectroscopy: Analysis of Copper Ore

Introduction:

Thousands of years ago, copper was abundant enough in quantity that it could be found on the Earth's surface. Prospecting for copper then was relatively simple. Recently, increased demand for copper resulted in numerous mines, and the search for copper ore is now very competitive. When a copper deposit is discovered, drilling is undertaken to determine the size and grade (concentration) of copper at the potential mining site.

Recently, two potential copper mines (labeled Sites A and B) were discovered in southeastern Arizona, just three miles east of Superior. The Resolution Copper Company conducted an exploration project in 2001 that indicates large deposits 7,000 feet below the surface. While the company is still in the early stages of developing the project, they want to determine which site (A or B) will be more profitable to mine. In order to determine this, the company has asked you to analyze samples from both sites for copper concentration. Based on your findings, the Resolution Copper Company will determine at which site they want to mine. Sites that are profitable to mine typically contain 0.4 - 1.0 % copper.

In this experiment, your group will analyze solutions made from copper ore extracted from both sites found by the mining company. In order to leach the copper from the ore and obtain a solution that can be analyzed, the solid ore sample was first dissolved in sulfuric acid and then filtered to remove sediments. Your group will analyze the resulting solutions from sites A and B to determine the concentration of copper at each site and determine if they are economically feasible to mine.

You will make solutions of known copper concentration as a baseline against which to compare your unknown samples from sites A and B. Your group will make diluted solutions of copper(II) sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) as standard solutions of known concentration. The relationship between concentration and absorption of the diluted solutions will be analyzed and the unknown samples' concentrations from sites A and B can be compared to the known solutions' concentrations.

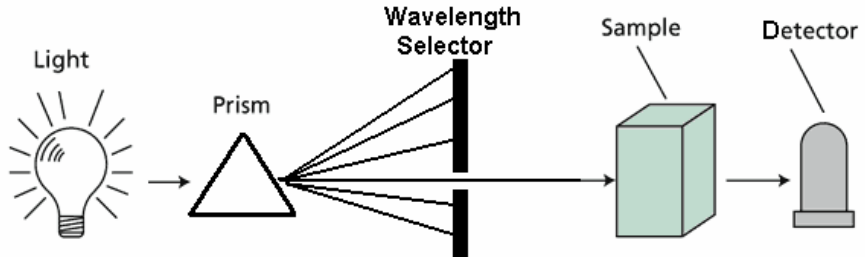
Spectrophotometric analysis is one method used to determine the concentration of a colored substance in solution. Concentration is determined by calculating the amount of light that passes through a sample and hits a detector. The more light that gets through, the less concentrated the solution is (fewer molecules of the colored substance that absorb light).

You have probably noticed this effect in everyday life if you have ever gotten a soda from a dispenser at a fast food restaurant and noticed that it was light in color. As soon as you noticed the light shade of the soda, you realized that it was low in syrup (or even missing syrup altogether). The light color of the drink immediately alerted you to the low concentration of syrup in the soda.

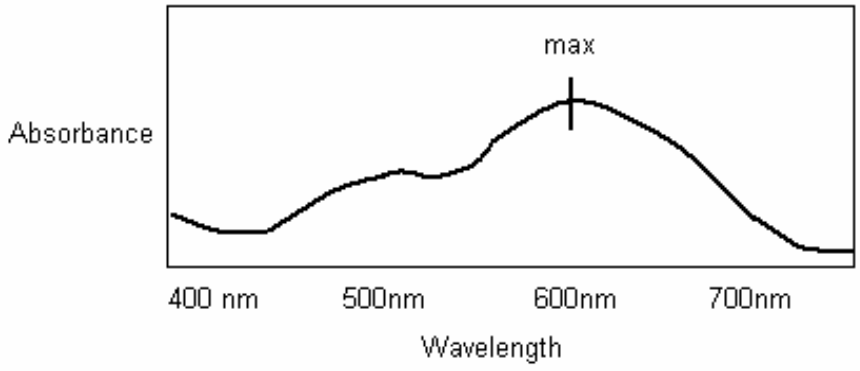
A solution containing a colored substance absorbs a specific color (or colors) of visible light and transmits the remaining colors. The amount of light a colored solution absorbs depends on the concentration of the colored substance (mg/mL of solution). Another factor affecting the amount of light absorbed is the color of light (wavelength in nm) selected. If a red solution is analyzed, you need to determine which color of light is best absorbed by the solution. If you select a wavelength in the red portion of the visible spectrum, what will happen to the light waves when they hit the red sample? (Hint: Is a red shirt absorbing red light or transmitting it?)

Spectrophotometric Analysis:

As discovered in the last lab, a spectrophotometer is an instrument that measures the intensity of a light beam of a given wavelength (color) that passes through a sample. Our machines use wavelengths between 325 and 1100 nm. Light of a given wavelength passes through the sample, hits the detector, and the instrument can calculate the **absorbance (A)** of the solution. Below is a schematic diagram of a typical spectrophotometer.



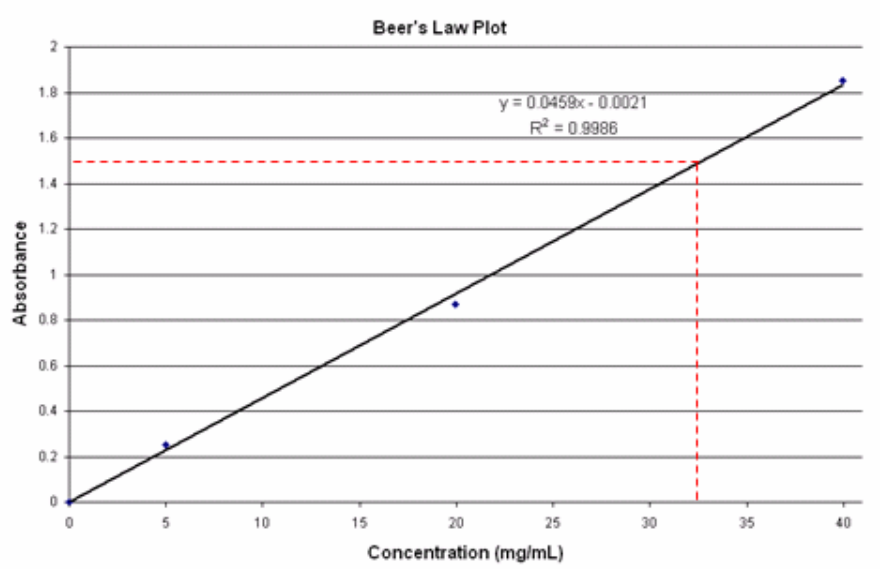
If the appropriate wavelength is not selected, the sample will not absorb enough light to make an accurate measurement. To select the best wavelength for measurements, we must find the wavelength of maximum absorption. To do this, we plot wavelength (x-axis) versus absorbance (y-axis) and simply find the wavelength that gives the maximum absorbance. The example below shows a peak absorbance occurring at about 610 nm.



Concentration may be expressed in several ways. In this exercise, it is expressed as the milligrams of copper per milliliter of solution (mg/mL). To determine the concentration of an unknown, a series of standards must first be prepared. Standards are made by diluting (adding water to) a stock solution to prepare solutions of **known** concentrations. The absorbance values and concentrations of these solutions are then graphed, and then the unknown concentration of a solution can be determined from the graph.

Beer's Law:

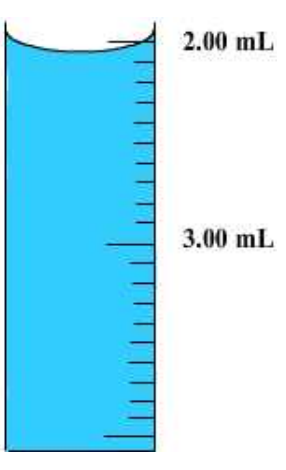
The relationship between concentration and absorbance can be summarized by using Beer's Law. Mathematically, Beer's Law states that these two quantities are directly (linearly) proportional. A plot of concentration (on the x-axis) versus absorbance (on the y-axis) will produce a linear relationship with a positive slope. A regression line representing the best straight-line fit of your data will produce an equation of the form **y = mx + b**. To determine the concentration of an unknown solution (for example, the amount of copper in an ore), the absorbance of the unknown solution can be measured and its concentration determined using the linear relationship from the graph. The plot on the next page shows an example of this relationship and the method for approximating the concentration of an unknown.



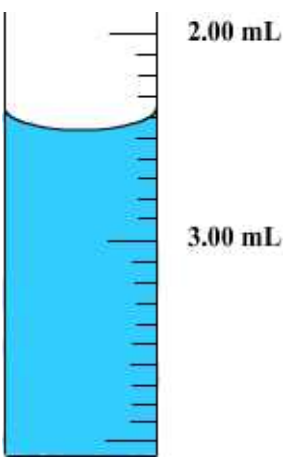
First, a regression line is plotted to give an equation of the line that best fits the data points. In this case, the equation is y = 0.0459 x – 0.0021. In this equation, *y* represents the absorbance value, 0.0459 is the slope, *x* is the concentration, and 0.0021 is the *y*-intercept. If an unknown is measured to have an absorbance value of 1.45, we can solve for concentration (x). Solving this equation for *x* with an absorbance (y value) of 1.45 gives a concentration value of 31.6 mg/mL.

You can check your calculation by estimating the concentration graphically. The concentration of the unknown can be estimated by finding the absorbance of the unknown solution, and a horizontal line (the red dotted line) can be drawn from the axis to the regression line of the data. At the intersection of the horizontal line and the regression line, a vertical line (red dotted line) is dropped down to the concentration axis. You can see from this estimation that the unknown has a concentration of approximately 32 mg/mL. This verifies that our calculation above is correct.

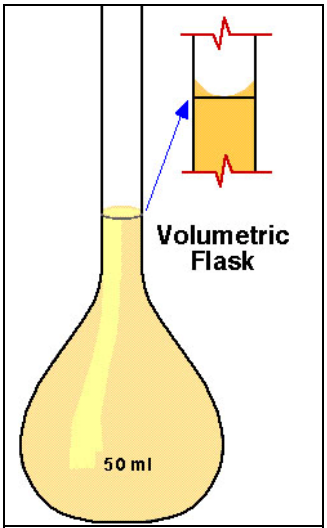
Using a Buret and Volumetric Flask:



2.06 mL of solution delivered



2.47 mL of solution delivered



50.00 mL Volumetric Flask

Reading a buret: Burets are designed to measure the amount of liquid that has been delivered. Therefore the volume begins closer to 0.00 mL and increases as you open the stopcock (parallel to the buret; the stopcock is **closed** when it is parallel to the ground). The graphics above show how a buret is read. Notice that this is the reverse of other glassware.

- You read the volume of liquid in a buret by reading from top to bottom. For example, the buret graphic on the left shows that the volume is 2.06 mL. The buret graphic on the right shows that the volume is now 2.47 mL. To determine the volume of liquid delivered, the initial volume is subtracted from the final volume (2.47 – 2.06; 0.41 mL were delivered).
- Volumes in burets are always read to **two decimal places**, the second decimal place is estimated. The long white lines indicate whole milliliters and the short white lines indicate tenths of a milliliter (0.1 mL). You can determine the whole and tenth milliliters and then estimate how far the meniscus is between tenths to estimate volume to the hundredths place.

Using a Volumetric Flask: Volumetric Flasks are calibrated to hold an exact volume of liquid when the bottom of the meniscus is exactly on the line. In this lab, you will use 50 mL volumetric flasks that measure volume to 50.00 mL. **If you add water above the line marked on the neck of the flask, you will have to remake your solution**. You cannot simply remove solution to get the volume down to the line. Fill water drop-wise using a disposable pipet to ensure that you do not pass the 50.00 mL mark.