SPECTROPHOTOMETRIC ANALYSIS OF COPPER

INTRODUCTION

This experiment has two purposes:

- To explore the technique of spectrophotometry, the use of light to determine some property of a substance.
- To determine the weight percent of copper in an unknown compound using spectrophotometry.

In the first part of the laboratory, you will explore a spectrophotometer, the instrument used in the laboratory. Then you will the conditions most suitable to use in the experiment. Finally, you will determine the concentration of copper in a solution, and from this information determine the amount of copper in an unknown sample.

In order to determine the concentration of copper in the unknown solutions we shall first construct a standard plot of the concentration of copper vs. the absorbance of solutions having known copper concentrations. According to Beer's Law, absorbance is directly proportional to concentration and so the resulting plot should be a straight line. This graph will be used to determine the concentrations of solutions containing known amounts of the unknown copper compound and from this we can determine the weight percent of copper.

EXPERIMENTAL PROCEDURE

Part A. Visible Spectrum

NOTE: This portion of the experiment may be done with a partner.

1. Take a piece of chalk that is about 2 cm long and rub it on the blackboard to produce a 45° edge or bevel on one end of the chalk.
2. Place the beveled piece of chalk into a test tube with the bevel pointed up, and put the test tube into the sample compartment of the Spec 20.
3. With the sample port open, look directly down at the piece of chalk and turn the sample tube until a small spot or slit of light is reflected off the bevel of the chalk. Adjust the light control knob until the light that is reflected off the bevel of the chalk is at maximum brightness.

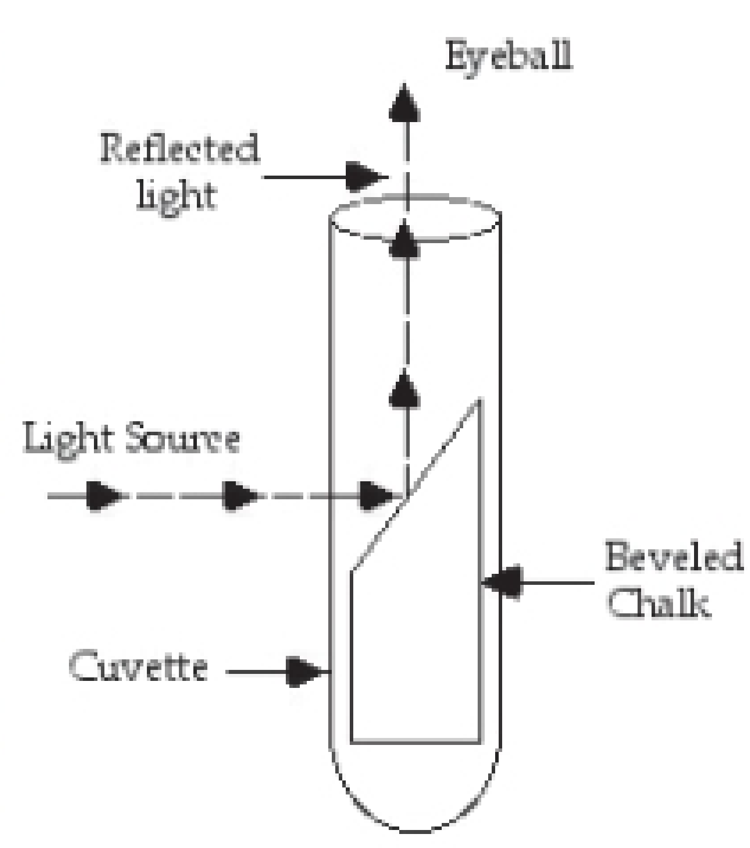

Figure A piece of beveled chalk in a small test tube allows you to see inside the sample container of the spectrophotometry and to observe the colors as a function of wavelength.

4. Turn the wavelength selector knob and notice how the color of the light reflected off the chalk changes color. By turning this knob, you can scan the entire visible spectrum.

Note that, although it is possible to find the absorbance of each of these solutions at any visible wavelength (~400 nm → ~700 nm), the Spec 20 gives most accurate results between 420 and 640 nm.

5. As you vary the wavelength, carefully note which wavelength ranges between λ = 400 to λ = 700 nm (where λ is the symbol for wavelength) correspond to which observed colors of the visible spectrum and record these wavelength ranges and colors in Table A in the Data Section of this write-up.
6. On Graph A provided in the Data Section, draw lines that correspond to the observed wavelength range for each primary color of the spectrum (red, orange, yellow, green, blue and violet) you recorded in Table A.

Part B. Light Absorption of Colored Solutions

In this portion of the experiment, two students may work as partners to prepare the calibration curve (using solutions 1-4 below). However, each student must do his or her own unknown solution (solutions 5 and 6).

1. Make sure the test tubes you are using are clean. Fill one with 0.24 M $CuSO_4$, one with 0.1 M $CoSO_4$ and one with distilled H_2O. Wipe off the outside of each test tube with a clean, dry cloth. Avoid fingerprints on the test tubes as you proceed with the experiment.
2. Starting at a wavelength setting of 420 nm, measure the absorbance (A) of both solutions at 420 nm. Record these values in Table B in the Data Section.
3. After zeroing the instrument at each different wavelength, measure the absorbance readings for both solutions at 20 nm increments from λ = 420 to at least 720 nm. (*For the copper containing solution, you will have to extend your measurements beyond 640 nm.)* Record absorbance readings for each of these solutions vs. wavelength in Table B in the Data Section.

Make sure you read the ABSORBANCE scale and not the transmittance scale on the instrument dial.

4. Plot the absorbance on Graph B in the Data Section. Make sure that you carefully record the different readings for the two different solutions.
5. When your measurements are complete, sketch a *smooth curve* connecting the points of each curve in order to depict the ABSORPTION SPECTRA of $CuSO_4$ and $CoSO_4$ respectively.

Part C. Concentration Effects

To determine the amount of copper in an unknown solution, you first have to explore the relationship between the concentration of copper ion and the amount of light absorbed by the sample. The important relationship is that the absorbance (A) of the solution is proportional to the concentration of the solution, c. That is, $A = \varepsilon \bullet \lambda \bullet c$, where ε is the molar extinction coefficient, a fundamental property of the molecules involved, and λ is the length of the sample cell. In this portion of the experiment you will confirm that relation and find out what the exact relationship is for your particular Spectronic 20 spectrophotometer.

1. Preparing Solutions:

Clean and dry the six 18 x 150 mm test tubes from your desk and label them 1 through 6. Four of these tubes will be used for solutions of known copper concentration and the other two will contain two different concentrations of the unknown copper compound. For each desk side (three people) you should have a set of two burets, one containing standard copper solution and one containing 1 M HNO_3. Use them to prepare the following solutions:

Test tube	Volume HNO_3	Volume Standard Copper	Remarks
1	10 mL	0	Reference standard for standardizing light control
2	6.0 mL	4.0 mL	Be sure to mix thoroughly
3	3.0 mL	7.0 mL	Be sure to mix thoroughly
4	0	10.0	
5	10.0 mL	0	Add about 0.3 g of unknown (accurately weighed). Be sure to mix thoroughly
6	10.0 mL	0	Add about 0.2 g of unknown (accurately weighted). Be sure to mix thoroughly

2. Measuring the Absorbance

When all six solutions have been prepared, obtain six spectrophotometer tubes, number them 1 through 6, and fill each tube about three-fourths of the way full with the corresponding solution.

a) For any species or mixture of species, the wavelength that is absorbed most strongly is abbreviated λ_{max}. Using Graph B prepared with the data from Part B, find λ_{max} for copper(II) sulfate, $CuSO_4$.

b) Set the spectrophotometer at λ_{max} for $CuSO_4$. ***(A wavelength of 645 nm is the recommended wavelength of this determination.)***

c) Standardize the Spectronic 20 and measure the absorbance of the solutions as described in *Using the Spectrophotometer* (and as outlined again below). Record the results in Table C2. Remember that you will calibrate and use the absorbance scale.

To adjust the left side of the absorbance scale leave the sample chamber closed and empty, and adjust the left hand knob on the front of the instrument panel until the absorbance scale reads infinity.

To adjust the right side of the absorbance scale to zero, first place your sample of pure HNO_3 (test tube #1) in the sample chamber. After closing the chamber use the light control knob (the right-hand knob on the front panel) to adjust the absorbance scale to zero.

After adjusting each end of the absorbance scale, place a test tube containing one of the copper-containing solutions in the sample chamber and read the absorbance from the scale. Repeat the adjustments of the left and right hand ends of the scale, and then once again determine the absorbance of the sample. Repeat at least one more time.

The actual amounts of the HNO_3 and $CuSO_4$ solutions used to prepare your samples should be recorded in Table C1.

Be sure that the tubes are clean. It is probably a good idea to rinse out each tube with a small amount of the solution before filling it with the solution. Be especially careful that there are no smudges or scratches near the bottom of the tube.

3. Handling the Data and Doing Calculations