Experiment 7A

ANALYSIS OF BRASS

MATERIALS: B & L Spectronic 20, 2 cuvettes, brass sample, 7 M HNO_3, 0.100 M $CuSO_4$, 2 M NH_3, two 50 mL beakers, 100 mL beaker, two 25 mL volumetric flasks, 100 mL volumetric flask, small watch glass, 5 large test tubes, 5 mL graduated pipet, 10 mL graduated cylinder, funnel, plastic dropper, test tube rack, steam bath.

PURPOSE: The purpose of this experiment is to determine the composition of a brass sample.

LEARNING OBJECTIVES: By the end of this experiment, the student should be able to demonstrate the following proficiencies:

1. Properly calibrate and use a Spectronic 20 spectrophotometer.
2. Determine the relationship between concentration and absorbance from data obtained from a Spectronic 20.
3. Convert between units of percent transmittance and absorbance.
4. Construct a calibration curve from solutions of known concentration.
5. Use a calibration curve to determine the concentration of an unknown solution.
6. Convert a molar concentration to a mass percent value.

DISCUSSION:

Brass is an alloy of copper and zinc metals. In this experiment, you will determine the mass percent of copper (and thus zinc) in a commercial sample of brass by employing a spectrophotometric method. Copper ions (Cu^{2+}) in solution will react with NH_3 to form the colored complex $Cu(NH_3)_4{}^{2+}$ which absorbs light in the visible region of the electromagnetic spectrum. The amount of light absorbed is proportional to the concentration of $Cu(NH_3)_4{}^{2+}$ and thus to the concentration of Cu^{2+}. This relationship, known as the Beer-Lambert Law, can then be used to determine the amount of copper in an unknown sample (refer to Appendix I).

The Beer-Lambert Law states that the absorbance (A) of a solution is proportional to the concentration of the absorbing species and the pathlength of light through the cell. This can be expressed as

$$A = \varepsilon \cdot l \cdot c$$

where ε is a proportionality constant (known as the molar absorptivity), l is the pathlength of light (in cm) passing through the solution in the cuvette, and c is the concentration of the absorbing species in moles/liter.

PROCEDURE:

Work with a partner to complete this experiment.

Part A. Preparation of the Unknown Brass Sample

1. Obtain a brass sample of unknown composition from your instructor.
2. Using an analytical balance, weigh a clean, dry 50 mL beaker. Record the mass in Table 1 of the Data and Analysis Section.
3. Using a top-loading balance, *pre-weigh* 0.10 to 0.12 g of the brass sample into your 50 mL beaker.
4. Using an analytical balance, weigh the beaker and brass sample. Record the mass in Table 1.
5. Label the beaker with your initials and place it in the hood. Carefully add 2 mL of 7 M HNO_3 measured with the graduated cylinder. Observe what happens.
6. Cover the beaker with a watch glass and place it on a steam bath (or hot plate set to low).

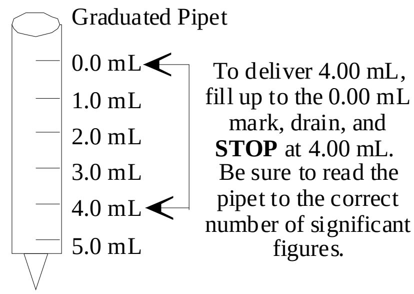
Answer Question #1 on page E7A-6. Each pair of lab partners may turn in one collaborative answer set.

7. While the brass is dissolving, proceed to Part B.

Part B. Preparation and Analysis of the Standard $Cu(NH_3)_4^{2+}$ Solutions

1. Using a graduated pipet, add the amount of 0.100 M $CuSO_4$ indicated in the table below to your 25 mL volumetric flask. Check the scale on the graduated pipet to see how to deliver the correct volumes.

Standard Solution	**Volume of 0.100 M $CuSO_4$** (added to 25 mL volumetric flask)
1	1.00 mL
2	2.00 mL
3	3.00 mL
4	4.00 mL
5	5.00 mL



2. **Using 2 M NH_3**, dilute the solution to the mark on the volumetric flask. Notice the formation of the deep blue $Cu(NH_3)_4^{2+}$ complex.
3. Mix the solution well by inverting and swirling the stoppered flask.
4. Transfer the prepared solution to a clean, dry, labeled test tube. Save this solution for analysis in Step 7.
5. Clean and rinse the volumetric flask and reuse it in the preparation of the next standard solution. Remember to dilute all solutions with 2 M NH_3 and mix well. Repeat this procedure until all of the standard solutions have been prepared. Since there are two 25 mL volumetric flasks per group, each person should prepare at least two of the required standard solutions.

Answer Question #2 on page E7A-6.

6. The instructor will demonstrate how to operate the spectrophotometer known as the Spectronic 20. Set the Spectronic 20 to the appropriate wavelength (λ). Record your selected wavelength in the Data Section. Calibrate the Spectronic 20 using the appropriate blank. In the Data Section, specify the blank you used.
7. Measure the percent transmittance (or absorbance) of each standard solution at your selected wavelength. Record the values in Table 2A.

Part C. Analysis of the Unknown Brass Sample (continuation of Part A)

1. Make sure your unknown brass sample from Part A is completely dissolved. Swirl the solution gently to check.
2. To the dissolved sample of brass, add 20 mL of 2 M NH_3 and carefully transfer this to a **100 mL** volumetric flask.
2. Thoroughly rinse the beaker several times with small amounts of 2 M NH_3, adding the rinses to the volumetric flask. This ensures that a complete transfer of the dissolved brass sample occurs. Fill the flask to the 100 mL calibration mark with 2 M NH_3. Mix the solution well by inverting and swirling the stoppered flask.
3. Measure the percent transmittance (or absorbance) of the unknown brass solution at the same wavelength you used in Part B and record this value in Table 3.

Clean up:

1. All solutions are dilute aqueous solutions and may be poured down the drain.
2. Wash your glassware. There should be no remnants of blue solutions in your flasks or test tubes.
3. Return all equipment to their original locations.

Name ______________________________ Section _____________________

Partner _____________________________ Date ______________________

DATA AND ANALYSIS SECTION

Experiment 7A

Part A. Preparation of the Unknown Brass Sample

Table 1

Mass of beaker	
Mass of beaker and brass sample	
Mass of brass sample	

Part B. Preparation and Analysis of the Standard $Cu(NH_3)_4^{2+}$ Solutions

Wavelength Selected = _______________ Blank Used = _______________________

Table 2A

Standard Solution	Measured % Transmittance
1	
2	
3	
4	
5	

Table 2B

Calculated Concentration of $Cu(NH_3)_4^{2+}$	Calculated Absorbance

Part C. Analysis of the Unknown Brass Sample

Table 3

Measured % Transmittance of unknown brass sample	
Calculated Absorbance of unknown brass sample	

DATA TREATMENT
Experiment 7A

Part B. Preparation and Analysis of the Standard $Cu(NH_3)_4^{2+}$ Solutions

(B.1) Calculate the concentration (mole/L) of $Cu(NH_3)_4^{2+}$ in Standard Solution 1. Show your work below. Record the result in Table 2B. Do similar calculations for Solutions 2 through 5 and record these results in Table 2B.

(B.2) From the data, calculate the absorbance of Standard Solution 1. Show your work below. Record the result in Table 2B. Do similar calculations for Solutions 2 through 5 and record these results in Table 2B.

(B.3) Using a spreadsheet program, construct a plot of absorbance versus concentration of $Cu(NH_3)_4^{2+}$ (data in Table 2B). Scale and label the axes appropriately. The data should exhibit a linear relationship, so plot the regression line (i.e., trendline) on the graph. Record the equation of the line and the R^2 value on the graph. Include the spreadsheet and graph (known as a *Calibration Curve*) with your lab report.

(B.4) Given that the solution in the cuvette has a thickness (or pathlength) of 1.0 cm, calculate the molar absorptivity (ε) of the blue $Cu(NH_3)_4^{2+}$ complex at your selected wavelength *using the information from your calibration curve and regression output*. Report ε with the correct units. Explain how you determined ε.

Selected wavelength, λ = ____________________

Molar Absorptivity of $Cu(NH_3)_4^{2+}$ complex (ε) = ________________________________

Part C. Analysis of the Unknown Brass Sample

(C.1) Using the calibration curve and regression output from Part B, determine the concentration of $Cu(NH_3)_4^{2+}$ in your unknown brass sample. Show how you determined this concentration.

Concentration of $Cu(NH_3)_4^{2+}$ = ___________________

(C.2) Calculate the mass of copper in your brass sample from the concentration of the $Cu(NH_3)_4^{2+}$ solution.

Mass of copper in brass sample = _________________

(C.3) Calculate the mass percent of copper and the mass percent of zinc in your original brass sample.

% Cu = _________________

% Zn = _________________

Name ________________________________ Section ______________________

Partner _______________________________ Date ________________________

IN-LAB QUESTIONS
Experiment 7A

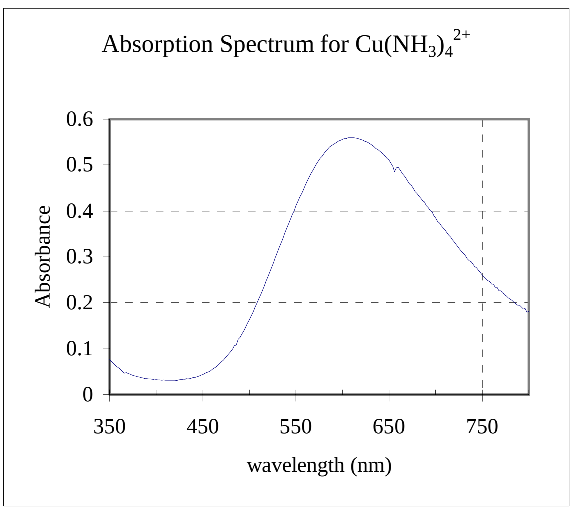
(Turn in this page before you leave lab.)

1. A toxic gas, NO_2, is released during the reaction of copper with nitric acid. Name this gas and describe it.

   NO_2 name = _____________________________

   Description of the gas released when Cu and HNO_3 react:

2. You will be using a spectrophotometer to determine the absorbance of the $Cu(NH_3)_4^{2+}$ complex in your solutions. However, you must first select the appropriate wavelength (λ) for this determination. Review the absorption spectrum for $Cu(NH_3)_4^{2+}$ below and select the best wavelength.



   a. What wavelength would you select? __________

      Explain your choice.

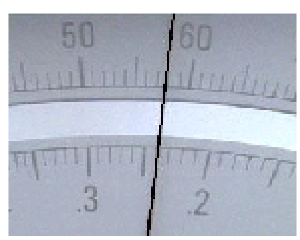
   b. In most spectrophotometric analyses, it is important to prepare a blank containing all reagents except the analyte (the species being analyzed). What is the appropriate blank for this experiment?

      ____

   c. To what decimal place should you read the % Transmittance scale on the Spectronic 20? ____________
      Read the scale below. Record all significant figures.

      % Transmittance = ________________

      (Note: if you have a Spect 20 that has a digital readout, you may read the absorbance value directly, vice the %T, recording all digits.)



← % T scale

   d. Convert your % Transmittance value in part c to an absorbance value. Abs = – log (%T/100)

      Absorbance = ______________________