Experiment 7A

8/7/18

ANALYSIS OF BRASS

MATERIALS: Spectronic 200 spectrophotometers, 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, 50 mL buret, 10 mL graduated cylinder, funnel, plastic dropper, test tube rack, hot plate.

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 200 spectrophotometer.
2. Understand the relationship between λ_{max} (wavelength of maximum absorbance) and the observed color of a solution.
3. Determine the relationship between concentration and absorbance from data obtained from a Spectronic 200.
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 a blue copper-ammine 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 path length 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 path length of light (in cm) passing through the solution in the cuvette, and c is the concentration of the absorbing species in moles/liter.

PRE-LAB:

Review Appendix I and section 4-8 in Kotz; complete the pre-lab exercises on p. E7A-6.
Graphing is required, so bring laptops to lab (with your goggles).

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 hot plate set **to low**.
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 buret, add the amount of 0.100 M $CuSO_4$ indicated in the table below to your 25 mL volumetric flask. Record the initial and final buret volumes in the table. (There is no need to refill the buret between uses.)

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. (There is no need to refill the buret between uses.) Since there are two 25 mL volumetric flasks per group, each person should prepare at least two of the required standard solutions.
6. The instructor will demonstrate how to operate the Spectronic 200 spectrophotometer. Refer to your prelab and set the Spectronic 200 to the appropriate wavelength (λ). Record your selected wavelength in the Data Section. Calibrate the Spectronic 200, using the appropriate blank, in "percent transmittance" mode. In the Data Section, specify the blank you used.
7. Measure the percent transmittance of each standard solution at your selected wavelength. Record the values in Table 2.

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 NH3 and carefully transfer this to the 100 mL volumetric flask.
3. Thoroughly rinse the beaker several times with small amounts of 2 M NH3, 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 NH3. Mix the solution well by inverting and swirling the stoppered flask.
4. Measure the percent transmittance 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

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Part A. Preparation of <u>100 mL</u> 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 2

Standard Solution	**Buret Readings**		**Measured Percent Transmittance**	**Calculated Concentration of $Cu(NH_3)_4^{2+}$**
	Initial	**Final**		
1				
2				
3				
4				
5				

Part C. Analysis of the Unknown Brass Sample

Table 3

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

(B.3) Using a spreadsheet program, calculate the absorbance of each solution from the percent transmittance. Use these values to construct a plot of absorbance versus concentration of $Cu(NH_3)_4^{2+}$. 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 from the graph, below. Include the spreadsheet and graph (known as a *Calibration Curve*) with your lab report.

Trend-line equation = ______________________________ R^2 = _________________

(B.4) From the trend-line above, and the Beer's Law equation, A = ε l c (where l = 1.0 cm), determine the molar absorptivity (ε) for the $[Cu(NH_3)_4]^{2+}$ complex at your selected wavelength, and record it below with the proper UNITS. Hint: ***y = mx + b***.

ε = ___________ (units:)

Part C. Analysis of the Unknown Brass Sample

(C.1) Calculate the absorbance of your sample from the measured percent transmittance value. 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.

$[Cu(NH_3)_4^{2+}]$ = __________________

(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 _____________ Date __________________________

PRE-LAB QUESTIONS
Experiment 7A

This lab requires graphing. Bring your laptop to lab.

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

   NO_2 name = ______________________________________

2. You will be using a spectrophotometer to determine the absorbance of the $Cu(NH_3)_4^{2+}$ complex in your solutions.

   a. According to the lab, what color will solutions of the copper-ammine complex, $Cu(NH_3)_4^{2+}$, be?

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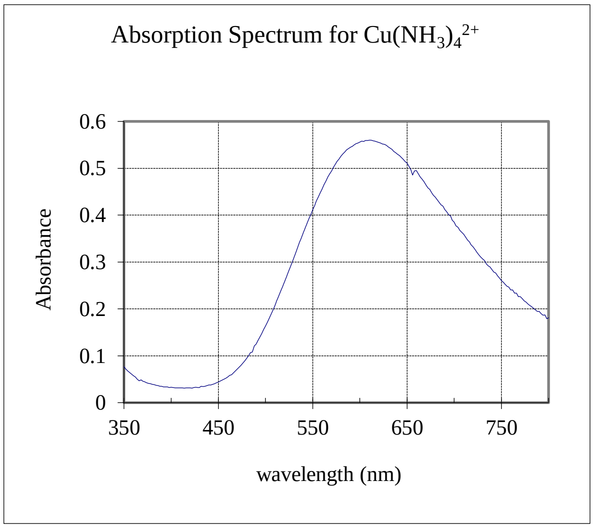
Examine the absortion spectrum for Cu $(NH_3)_4^{2+}$ (aq) solutions.



b. What wavelength should be selected for measuring the absorbance of the copper – ammine solutions? _______________

Explain your choice.

c. How is the wavelength you selected in part b related to the observed color of the $Cu(NH_3)_4^{2+}$ solution? (Review Appendix I for help.)

3. In most spectrophotometric analyses, it is important to prepare a blank containing all reagents <u>except</u> the analyte (the species being analyzed). Refer to the sample preparation instructions in Part B of this experiment to determine the appropriate blank for this experiment.

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4. In this lab, you will prepare a series of dilute solutions, measure the absorbance of each, and plot a calibration curve of absorbance vs. concentration. Calculate the concentration (Molarity) of a solution made by diluting 3.00 mL of 0.100 $CuSO_4$(aq), with NH_3(aq), to the 25-mL mark of a volumetric flask.