Colorimetric Determination of Cu Content in Brass

Brass is an alloy of copper and zinc. The mass percentage of copper in brass can be determined by first reacting it with concentrated nitric acid, and then using a colorimeter to analyze the color intensity of the copper(II) nitrate solution that forms.

Copper reacts readily with oxidizing agents. In this experiment, concentrated nitric acid oxidizes copper metal to the copper(II) ion. The unbalanced equation is:

$$\text{Cu(s)} + \text{HNO}_3(\text{aq}) \rightarrow \text{Cu(NO}_3)_2(\text{aq}) + \text{NO}_2(\text{g}) + \text{H}_2\text{O(l)}$$

As products of this reaction, copper(II) nitrate is a water-soluble salt that produces a blue solution, and nitrogen dioxide, NO$_2$, is a dense, toxic, red-brown gas. The zinc from the brass forms zinc nitrate solution, which is colorless.

A solution of copper(II) nitrate appears blue because when white light passes through the solution, the dissolved copper ions absorb the red components of the light, while transmitting the blue portions of the light. The instrument we will use is a colorimeter, a spectrometer which uses light in the visible region.

Spectroscopic analytical methods rely on the ability of substances to absorb (or emit) electromagnetic radiation. The figure below shows the basic components of a spectrometer. The spectrometer measures the amount of light transmitted by the sample by comparing the intensity of the light emitted from the light source with the intensity of the light that emerges from the sample. As the concentration of the absorbing species increases and its color becomes more intense, the amount of light transmitted by the solution increases, causing less light to reach the detector.

![Spectrometer diagram](image)

The ratio of the intensity of the transmitted light, $I_t$, to that of the incident light, $I_o$, is called the transmittance, $T$, of the electromagnetic radiation by the sample. This ratio, expressed as percent, is

$$\frac{I_t}{I_o} \times 100 = \% T$$

Incident light, $I_o$, and transmitted light, $I_t$, for a sample of thickness $b$:

Most colorimeters have a %$T$ (percent transmittance) scale. However, chemists often perform calculations based on the amount of light absorbed by the sample, rather than the amount of light transmitted, because absorption is directly proportional to the concentration of the absorbing substance. Absorbance is related to transmittance:

$$A = - \log T \quad (\text{where transmittance, } T, \text{ is expressed as a fraction, not as } \%)$$
The relationship between absorbance and transmittance is shown:

Beer’s law relates the amount of light being absorbed to the concentration of the substance absorbing the light:

\[ A = a b c \]

In this equation, \( A \) is the measured absorbance, \( a \) is the molar absorptivity constant (a characteristic of the substance being monitored), \( b \) is the path length through which the radiation must pass, and \( c \) is the molar concentration of the absorbing substance. Thus, the concentration is directly proportional to absorbance.

In this lab, you will generate a calibration curve for copper(II) nitrate absorbance. You will prepare four \( \text{Cu(NO}_3\text{)}_2 \) solutions of known concentration (standard solutions). When a graph of absorbance vs. concentration is plotted for the standard solutions, you should see a linear relationship. The concentration of the unknown \( \text{Cu(NO}_3\text{)}_2 \) solution is then determined by measuring its absorbance with the colorimeter and using your copper(II) nitrate absorbance curve.

**Materials**

- brass .22 caliber shells
- 100-mL beaker
- 100-mL volumetric flask
- assorted volumetric pipets
- test tubes
- conc. HNO\(_3\)

Make sure to record in your data section the number on the Colorimeter, the power adapter and the power cord.

**Procedure:**

1. Determine the mass of two brass .22 caliber rifle shells to \( \pm \) 0.01 g. Place the shells in a 100-mL beaker. Under the fume hood, add about 10 mL of 15.8 M HNO\(_3\) (aq). **Caution: the reaction is very vigorous and the red-brown gas evolved, NO\(_2\), is toxic!!!** After the metal dissolves completely, add 50 mL of distilled water to the beaker. Transfer the solution to a 100 mL volumetric flask and dilute to 100.0 mL.

2. You will need ~4 mL to fill the cuvette. Obtain 10-11 mL of 1.00 M \( \text{Cu(NO}_3\text{)}_2 \) (aq) stock solution in a 10 mL graduated cylinder. Use a volumetric pipet to transfer 10.00 mL of this stock solution into a clean test tube and, using a volumetric pipet, add 10.00 mL of distilled water. **Thoroughly mix the solution.** This makes 20.00 mL of 0.500 M \( \text{Cu(NO}_3\text{)}_2 \) (aq). Perform additional dilutions to make 10.0 mL each of 0.100 M, and 0.050 M \( \text{Cu(NO}_3\text{)}_2 \) (aq). Use your own diluted samples to make further serial dilutions.

3. Calibrate the colorimeter. First “zero” the colorimeter for distilled water.
   a. Turn on. Press Select and Start/Stop simultaneously.
   b. Fill a cuvette with distilled water, holding only on the ridged sides. Insert the distilled water reference so the ridged sides are facing top and bottom.
   c. Push Select. Colorimeter should read Orange 630 nm; this is the wavelength we will use to analyze the solutions. If the display indicates another color, press select until you find Orange.
   d. Press Start. Colorimeter should read 100% Transmittance. If not, press Stop and try again.

4. Determine the calibration curve for \( \text{Cu(NO}_3\text{)}_2 \) using your 0.0500M, 0.100M, and 0.500 M solutions. Use the same cuvette for all solutions; start with the most dilute sample (why?) and rinse cuvette with each solution being tested before measuring absorbance.

5. Determine the percent transmittance of your dissolved brass shell.
Analysis

1. Prepare a data table with your calibration samples and brass analysis data. Show both % transmittance and absorbance.

2. Construct a graph of Absorbance vs. Concentration for you standard copper nitrate solutions. Calculate the linear regression (best-fit line) equation of absorbance vs. concentration. (Be sure to paste in your graph.)

3. Using your calibration curve and the absorbance of your brass shell solution, estimate the concentration of copper(II) nitrate. Calculate the wt% copper in your brass. Assume the class average for wt% copper in brass is the “true value”. Calculate your % error; explain any errors greater than 5%.