

## Lab 12.4

# Percentage of Copper in Brass

### BACKGROUND

Measuring how much of which wavelengths of light are absorbed by a substance and getting useful information about the substance from the results, is the scientific discipline of spectroscopy. The visible spectrum is the only part of the electromagnetic spectrum that we can access with equipment found in a typical school chemistry laboratory. The basic principles of spectral analysis that you learn in high school can also be applied to the more sophisticated instrumentation required to access the ultraviolet, infrared, and x-ray regions. What you learn by performing this lab will help you to understand more sophisticated instruments that you may encounter in a college or university.



In a visible spectrophotometer, we shine a beam of light into a solution containing the sample and detect how much of it comes out of the other side of the solution. By comparing the amount of light transmitted by the pure solvent to the amount transmitted when the sample is dissolved in it, we can calculate a quantity called the *absorbance*. Spectrophotometers can report measurements as percent transmittance (%T) or directly as absorbance. In the investigation, you will be guided to discover the relationship between transmittance and concentration and, ultimately, the relationship between transmittance, absorbance, and concentration of solution.

Spectrophotometry is an extremely important tool used in forensic science to determine the detailed chemical composition of evidence obtained from a crime scene. It can be used to determine the concentration of either a single chemical species in solution or even the concentration of a species within a mixture of species in solution. For example, it can be used to determine the mass percent of copper in brass shell casings collected by the crime scene investigator (CSI), and the match the brass composition to a particular manufacturer.

**XRF (X-ray fluorescence spectrometry) analysis of cartridge case brass -- Note: numeric values are mass %**

	CU -copper	ZN -zinc	FE -iron	Si -Silicon	Cr - Chromium	Alloy Series	Alloy Name	Vintage	Cartridge
Norma	77	23	0.15			C24000	Low Brass	2009 lot	6mmBr
Lapua	75	25	0.16	0.19	0.08	C24000	Low Brass	2010 "Blue Box"	6mmBr
Lapua	62	36	0.26			C28000	Muntz Metal	2010 "Brown Box"	6mmBr
Remington	80	20	0.12			C24000	Low Brass	2009 lot	22-250
Winchester	72	27		0.53	0.19	C26000	Cartridge Brass	2009 lot	308WIN
Federal	78	20	0.15		0.17	C24000	Low Brass	2009 Lot	308WIN
S & B	72	28	0.17	0.15		C26000	Cartridge Brass	2009 lot	9x19mm

### Safety

- **Nitric acid (HNO<sub>3</sub>) is highly corrosive to skins and eyes.**
- The gas evolved as the casing is dissolved in nitric acid is NO<sub>2</sub>. NO<sub>2</sub> is a significant irritant to mucous membranes, eyes, throat and lungs. The entire reaction of the brass with the nitric acid must be completed in the fume hood.
- Copper (II) nitrate is an eye and lung irritant and may be toxic if ingested. It reacts violently with strong oxidants and decomposes to form toxic substances when heated strongly.
- Spills should be neutralized and cleaned up immediately. Any solutions that contact skin should be rinsed off with plenty of water.
- Goggles and aprons must be worn.

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

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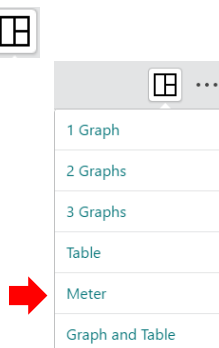
### Using the GoDirect Colorimeter

1. Plug the GoDirect colorimeter into an available USB port on the laptop.
2. Log into the laptop computer and open the “Vernier Graphical Analysis” application from the desktop.
3. Calibrate the colorimeter.
  - a. Prepare a *blank* by filling an empty cuvette 3/4 full with distilled water. Seal the cuvette with a lid.

#### Working with Cuvettes

- All cuvettes should be wiped clean and dry on the outside with a tissue before inserting into the colorimeter.
- Handle cuvettes only by the top edge of the ribbed sides.
- All solutions should be free of bubbles. Tap on table top to remove bubbles.
- Always position the cuvette with its reference mark facing toward the white reference mark at the top of the cuvette slot on the Colorimeter.

- b. Place the blank in the cuvette slot of the Colorimeter and close the lid.
  - c. Press the < or > button on the Colorimeter to set the wavelength according to the pre-lab questions. Then calibrate by pressing the CAL button on the Colorimeter. When the LED stops flashing, the calibration is complete.
  - d. Empty the water from the cuvette and set aside to dry.
4. Click on the upper right corner “Untitled”. 
    - a. Select “New Experiment” from the menu.
    - b. Select “Sensor Data Collection”
    - c. Click on Graph button in upper right corner. 
    - d. Select “Meter”
    - e. Collect data.



## Percentage of Copper in Brass

### I. PURPOSE

To determine the percentage of copper in a brass sample to identify its manufacturer.

### II. MATERIALS

- |   |                       |                               |
|---|-----------------------|-------------------------------|
| 1. Colorimeter                                | 5. Distilled water    | 9. Syringes                   |
| 2. Cuvettes with covers                       | 6. Used bullet casing | 10. Balance (0.001 precision) |
| 3. 0.40 M $\text{Cu}(\text{NO}_3)_2$ solution | 7. Test tubes (4)     | 11. 100mL volumetric flask    |
| 4. Concentrated (15.8 M) $\text{HNO}_3$       | 8. Test tube rack     |                               |

### III. PROCEDURES

A. Prepare the solutions for analysis.

- Obtain a bullet casing and examine it carefully. If there are any readable markings on the casing record them for future reference. Get the mass of the bullet casing to the nearest 0.001 g.
- Take the bullet casing to the fume hood, place it in a beaker and add the volume of concentrated  $\text{HNO}_3$  calculated in the Pre-Lab questions plus 2mL. If the casing is not completely covered by the acid, add just enough acid to cover it. Leave it in the fume hood until ready to use.
- Using the syringes, prepare the dilutions needed for the standard curve from the 0.4M standard cupric nitrate.
- Prepare the colorimeter for calibration according to the instructions in the Background section.

B. Calibrate the colorimeter.

*Follow the guidelines for using a colorimeter listed in the background section.*

- Prepare a *blank* by filling an empty cuvette 3/4 full with distilled water. Seal the cuvette with a lid.
- Place the blank in the cuvette slot of the Colorimeter and close the lid.
- Press the < or > button on the Colorimeter to set the wavelength to a value close to the one calculated in the prelab questions. Then calibrate by pressing the CAL button on the Colorimeter. When the LED stops flashing, the calibration is complete.
- Empty the water from the cuvette and set aside to dry.

C. Determine the standardization curve.

- Using the lowest of your concentrations, rinse a cuvette twice with ~1 mL amounts and then fill it 3/4 full. Place the cap on the cuvette and wipe the outside with a tissue, place it in the colorimeter, and close the lid.
- When the value displayed on the screen has stabilized, record the absorbance value in your data table.
- Repeat Steps C.1 & C.2 with each of the remaining solutions in order of increasing concentration (including the stock 0.4M solution).

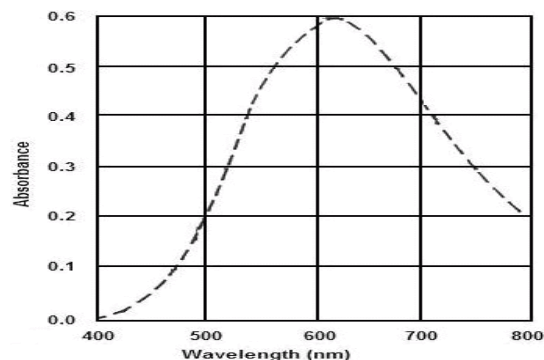
D. Analyze the casing solution.

- Remove the beaker from the fume hood and transfer the solution to a 100 mL volumetric flask. Rinse the beaker 3 – 4 times with 5 mL of distilled water and add the washings to the flask. Dilute to the final volume of 100.0mL. Use a wash bottle as you get close to the mark on the flask, so you don't overshoot the line.
- Rinse a cuvette twice with the unknown solution and fill it about 3/4 full. Place the cap on the cuvette and wipe the outside with a tissue, place it in the colorimeter, and close the lid.
- When the value displayed on the screen has stabilized, record the absorbance value in your data table.
- Discard the solutions as directed by your instructor.

### IV. PRE-LAB QUESTIONS

- At right is a graph of absorbance vs. wavelength for the  $\text{Cu}^{2+}$  ion. What wavelength should you choose to measure the copper concentration spectrophotometrically? Why?
- Below is some absorbance data for a solution of  $\text{Cu}^{2+}$ . Use the best-fit line for this data to determine the concentration of a solution with an absorbance of 0.48. Write the equation of the line and show your work to find the concentration.

Concentration (M)	0.20	0.30	0.40	0.50
Absorbance	0.27	0.41	0.55	0.69



- You will need to make four dilutions of the 0.40 M  $\text{Cu}(\text{NO}_3)_2$  stock solution. Determine the volume of  $\text{Cu}(\text{NO}_3)_2$  stock solution and volume of  $\text{H}_2\text{O}$  needed to make 10.00 mL of four solution with concentrations between 0.400 M and 0.050 M. Try to space the concentrations out evenly over this range. Show all work.

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4. Assuming your bullet casing is 100 percent copper by mass, calculate the minimum volume of concentrated (15.8 M)  $\text{HNO}_3$  needed to dissolve the casing. The reaction is:



5. Suppose a solution was too concentrated for an accurate reading with spectrophotometer. The concentrated solution was diluted by placing 1.00 mL of the concentrated solution in 4.00 mL of water. The solution was then placed in the spectrophotometer, an absorbance was obtained, and after a few calculations the molar concentration was calculated to be  $3.5 \times 10^{-6}\text{M}$ . What was the concentration of the original solution before dilution?

### V. DATA & CALCULATIONS

#### A. DATA

Data Table

	Concentration (M)	Absorbance
Trial 1		
Trial 2		
Trial 3		
Trial 4		
Standard Solution	0.40	
Unknown	???	
Mass of Casing (g)		

#### B. CALCULATIONS

1. Enter the data from your table into a graphing calculator and find the linear regression. The linear-regression statistics for these two data columns are displayed for the equation in the form

$$y = mx + b$$

where  $x$  is concentration,  $y$  is absorbance,  $a$  is the slope, and  $b$  is the  $y$ -intercept. (*Note: One indicator of the quality of your data is the size of  $b$ . It is a very small value if the regression line passes through or near the origin. The correlation coefficient,  $r^2$ , indicates how closely the data points match up with (or fit) the regression line. A value of 1.00 indicates a nearly perfect fit.*)

Record the equation of the line (in scientific form) and your  $r^2$  value.

2. Use the equation of your line to determine the concentration of  $\text{Cu}^{2+}$  ion in the 100 mL solution.  
3. Calculate the mass of copper present and the percent by mass of copper in your bullet casing.

### VI. POST-LAB QUESTIONS

1. Use the material provided at the end of the Background section to determine the manufacturer of the bullet casing. Does this agree with any markings you were able to read on Day 1?  
2. Data is given below for the absorbance of  $\text{CuSO}_4$  vs. its concentration. What mass of copper could be produced by a complete reaction of excess zinc metal with 50.0 mL of a  $\text{CuSO}_4(\text{aq})$  that has a measured absorbance of 0.685? Show all work.

Concentration (M)	0.08	0.16	0.24	0.32	0.40
Absorbance	0.186	0.372	0.587	0.753	0.955

### VII. CONCLUSION