Using a Buret and Volumetric Flask:

Reading a buret: Burets are designed to measure the amount of liquid that has been delivered. Therefore the reading (also called “volume”) begins at 0.00 mL and increases as you open the stopcock (parallel to the buret; the stopcock is closed when it is parallel to the ground). The graphics above show how a buret is read. Notice that this is the reverse of other glassware.

- You read the buret from top to bottom. For example, the buret graphic on the left shows that the reading is 2.06 mL. The buret graphic on the right shows that the reading is now 2.47 mL. To determine the volume of liquid delivered, the initial volume is subtracted from the final volume \((2.47 - 2.06; 0.41 \text{ mL were delivered})\).

- Burets are always read to two decimal places, the second decimal place is estimated. The long white lines indicate whole milliliters and the short white lines indicate tenths of a milliliter (0.1 mL). You can determine the whole and tenth milliliters and then estimate how far the meniscus is between tenths to estimate volume to the hundredths place.

Using a Volumetric Flask: Volumetric Flasks are calibrated to hold an exact volume of liquid when the bottom of the meniscus is exactly on the line. In this lab, you will use 50 mL volumetric flasks that measure volume to 50.00 mL. If you add water above the line marked on the neck of the flask, you will have to remake your solution. You cannot simply remove solution to get the volume down to the line. Fill water drop-wise using a disposable pipet to ensure that you do not pass the 50.00 mL mark.
**Procedure:**

**Part I: Preparing the Standard Solutions**

**Lab Notebook**

Note: Anytime you make a table, it should have a title describing the data within the table.

Your table for Part I should have the following column headings:

- “Solution”
- “Starting buret reading (mL)”
- “Ending buret reading (mL)”
- “Total delivered (mL)”
- “Concentration (mg/mL)” (Note: Calculations should be completed in lab notebook)

Your table should have a row for each solution to be made. For example: “0mL solution”, “1mL solution”, “2mL solution”, etc. should be used in the “Solution” column. Note that for the 0mL solution, there will be no buret measurements since it contains no CuSO₄. Place an “X” in the boxes for the buret measurements for 0mL.

Turn on the Genesys Spec-20 before you begin this part so it will have plenty of time (about 15 minutes) to warm up.

You will work in groups to prepare solutions for this experiment. Your group will be provided with a stock copper solution of copper (II) sulfate, CuSO₄, in a buret. Record its concentration (in mg/mL) on your report sheet. It is important that all students learn how to use the buret and spec-20. Each student should prepare at least one solution (Part I) and take at least 1 absorbance reading (Parts II and III).

Your group will make 5 standard solutions (using approximately 1 mL, 2 mL, 4 mL, 6 mL, and 8 mL of stock solution).

1) Use the buret of stock copper solution to deliver volumes as close to whole numbers as you can measure. Add each volume of solution to a clean 50.00 mL volumetric flask. Record the exact volume of stock solution added. Add deionized (DI) water from your wash bottle to the solution. When the meniscus gets close to the line, you may use the plastic pipet to add water drop-wise. Be sure the bottom of the meniscus does not go above the line. If it does, you will have to remake that solution! Stopper the flask and invert it several times to thoroughly mix the solution.

2) Repeat this process to make the next 4 solutions. Label your flasks with pencil or tape. Deliver approximately 1 mL, 2 mL, 4 mL, 6 mL, and 8 mL of stock solution, and fill each volumetric flask to the line with deionized water. Remember to record the exact volume of the buret before and after delivering the stock solution.

3) Stopper all your labeled flasks, invert them several times to thoroughly mix the solutions, and set them aside for part III.
**Using the Spec-20:**

**Part II: Finding the Maximum Absorbance for Copper Solutions**

****Lab Notebook**

Your table for Part II should have following column headings:
- “Wavelength (nm)”
- “Absorbance”

Your table should have a row for each wavelength to be tested

- Hint: You will be taking measurements initially from 400-900nm in increments of 100nm. After finding the wavelength which has highest absorbance, you will then take readings 10, 20, and 30nm above and below the wavelength with the highest absorbance. From this information, how many rows should you have?

- Below your table, record the wavelength which gave you the maximum absorbance, this is called $\lambda_{\text{max}}$ (lambda max). Your instructor must sign off on this wavelength before you move to the next part.

- You will then graph all your data on the graph paper provided in the worksheet.

Your group will generate a table and graph of wavelength versus absorbance to determine the wavelength at which the maximum absorbance occurs for your 8.00 mL solution. As you take your readings, you will have to recalibrate the Spec-20 (“set the blank”) using DI water for each new wavelength.

1) Press the “A / T / C” button until an “A” appears in the upper right corner of the display.
2) Use a disposable pipet to fill one cuvette 2/3 full with DI water and a second cuvette 2/3 full with your last (8.00 mL stock) solution. Check each cuvette for air bubbles. If you see air bubbles clinging to the walls of a cuvette, tap the cuvette gently to get the bubbles to rise to the top of the solution and escape. Use a “Kim-wipe” to wipe off the outside of the cuvettes.
3) Follow steps a – e below to determine the absorbance for each wavelength.
   a) Place the water cuvette in the holder with the clear sides aligned with the arrow. (This arrow shows the direction in which light is aimed.)
   b) Select the first wavelength to test (400 nm) using the up/down nm buttons. (They scroll quickly if you hold the button down.)
   c) Press the “0 Abs/100 % T” button to calibrate the instrument for that wavelength.
   d) Remove the cuvette with water and replace it with the cuvette of solution. DO NOT press any buttons!!!
e) Read the absorbance on the display for your solution. If it reads a small negative number, record a value of 0 for that reading.

5) Repeat steps a – e for water and the first solution cuvettes at a wavelength of 500 nm. Record the absorbance values in the table under Part II on the Report sheet. Also repeat steps a-e to obtain absorbance readings at wavelengths of 600, 700, 800, and 900 nm.

6) Examine your absorbance readings to find the maximum value for the 100-nm intervals. Take readings 10, 20, and 30nm above and below the wavelength with the maximum absorbance. For example, if you found 600 nm to be the highest value, repeat steps a – e for wavelengths of 610, 620, 630, 590, 580, and 570 nm.

6) Plot and connect the 12 data points on the graph provided in the handout. The wavelength that gives the maximum absorbance, $\lambda_{\text{max}}$ (lambda max), is the wavelength you want to use for Part III of this experiment.

7) Have your instructor sign off on your chosen wavelength before you continue to Part III.

**Part III: Measuring Absorbance Values for the Known and Unknown Solutions**

<table>
<thead>
<tr>
<th><strong>Lab Notebook</strong></th>
</tr>
</thead>
</table>

Your table should have the following column headings:

- “Solution”
- “Concentration (mg/mL)” (same values from your first table for your known solutions)
- “Absorbance” (for standards and unknowns)

You should have a row for each solution to be tested. For example: “1mL solution”, “2ml solution”, “Site A”, etc. should be used in the “Solution” column. For the 0mL sample, you should record a value of 0 for the absorbance.

Your group will use the wavelength found in Part II to determine the absorbance for each solution made in Part I. You will then analyze the copper content in unknown samples taken from the two drill sites (A and B).

1) Use a cuvette filled 2/3 full with deionized water to calibrate the Spec-20. Select the appropriate wavelength found in part II. Wipe the cuvette off with a clean “Kim-Wipe”, and place the water cuvette in the sample holder. Close the lid, and press the 0 Abs/100% T button to set the absorbance of the water sample to zero. **Do NOT press this button again during Part III.**

2) Empty the cuvette, and rinse it twice with small amounts of the first solution to be used. Fill the cuvette 2/3 full with that solution. Wipe the cuvette off with a “Kim-Wipe” before placing it in the sample holder. After closing the lid, read and record the absorbance of that solution. **Do not change the wavelength or press any other buttons on the top of the Spec-20 while you obtain absorbance readings for the standard solutions!**
3) Repeat step 2 for all of the standard solutions you made in Part I.
4) **Save the solutions until all data collection and graphing is complete.**
5) The unknowns contain the same colored metal (copper) solutions as the standards you prepared, but at different concentrations. You will analyze your two un**diluted** unknowns in the same manner as the standards.
6) **Each group** should obtain solution from Site A and solution from Site B. The unknowns are in dropper bottles. Add enough solution from the Site A bottle to fill the cuvette 2/3 full. Obtain one unknown at a time to avoid mixing up the samples. **Do NOT dilute the unknowns!**
7) Without pressing any buttons on the spectrophotometer, place your cuvette with the unknown from Site A in the spectrophotometer and record its absorbance reading. Repeat with your unknown from Site B.

**Waste Disposal:** After you have recorded and graphed all of your concentration and absorbance data from your solutions, discard them in the waste container in the fume hood.

**Calculations:**

**Concentration of standard solutions:**
Determine the concentration of dye in each of your standard solutions by using the dilution formula: $M_1 V_1 = M_2 V_2$ where:

- $M_1$ is the initial concentration of the stock solution (mg/mL)
- $V_1$ is the total volume that you delivered from the buret for each solution (mL). Use the exact volume delivered, not just 8.00 mL.
- $M_2$ is the final concentration of each standard solution you prepared (mg/mL) This is what you are trying to calculate for each solution.
- $V_2$ is the total volume of each solution (mL). This will be the same for all of your solutions. What total volume of solution was used for each standard?

**Graphs:**
You will use Excel to enter and graph the data you obtain in this lab. The following instructions are for Excel 2007.

- Enter your data in the worksheet. Enter exact concentrations in the first column and absorbance values in the second. Be sure to include the origin (0, 0) as a data point (from the calibration of water) for a total of 6 data points.
- Highlight the two columns of data
- Select: **Insert ➔ Scatter.** Select the scatter plot with data points only.
- Select the graph. From the menu, select: **Layout.** From here, you can add at title to your graph and label your axes (Concentration on the x-axis (units of mg/mL) and absorbance on the y-axis (unitless)).
- Add a regression line to your data points to find the best fit of a linear equation to your data. Right-click one of the data points in your graph. Select “Add Trendline”. For the type of trendline, choose “Linear” and check the boxes for “Display equation on chart” and “Display R-squared value on chart”.
- The equation option will provide the equation for the line ($y = mx + b$) that best fits
your data points. This line will be used to calculate the concentration for your unknown solutions.

- The R-squared value will indicate how close your data points are to a straight line. A value close to 1.00 indicates your data is very close to linear.
- You may want to clean up the formatting of the graph before printing. You can right-click the background to clear it. You can also select and delete the label to the right of the graph.
- Print your graph, have your instructor initial it, and turn in a copy with your lab report.

Use the linear equation \( y = mx + b \) to determine the concentration of copper in the two unknown solutions.

**Calculations using linear regression equation:**

The general equation for a line is represented as \( y = mx + b \), where \( y \) is the variable on the y-axis (the dependent variable), \( m \) is the slope of the line, \( x \) is the variable on the x-axis (the independent variable), and \( b \) is the y-intercept.

- In your lab notebook, include the equation of the linear regression line
- Also include the \( R^2 \) values for the line (\( R^2 \) value of 1 means it is a perfect line).
- Use the equation from the regression line to find the concentration of both unknown solutions (Site A and Site B).

Make sure to show your calculation in your lab notebook. (Hints: Which variable(s) in the linear equation do you know? Which variable are you solving for?)
**Introduction to Spectroscopy: Analysis of Copper Ore: Lab Report**

Name: ___________________________  *Turn in pages 7-9 with your lab notebook copies*

Section: _________________________

**Part I: Preparing the Standard Solutions, Part II: Finding the Maximum Absorbance for Copper Solutions and Part III: Absorbance Values for Known and Unknown Solutions:**

A statement of the purpose of the experiment, description of the procedure you follow, all data tables, all calculations, a discussion of the results and a conclusion should be in the lab notebook.

**Part II: Finding the Maximum Absorbance for Copper Solutions:**

Using the data you collect from Part II, plot all of your data in your notebook. Connect the points with a smooth curve. *Neatly recreate the graph here for your instructor.*
Questions
1) What is the name of the symbol $\lambda_{\text{max}}$? What is the definition of this term? Which graph allowed you to determine $\lambda_{\text{max}}$?

2) Just like the Cu$^{2+}$ ion used in today’s lab, some other metal ions have a distinctive color when in an aqueous solution. Some examples of ions that give colors in aqueous solution are: Cr$^{3+}$, Ni$^{2+}$, and Co$^{2+}$. However, other metallic ions such as Mg$^{2+}$, Al$^{3+}$, and Ti$^{4+}$ are relatively colorless.

   a. Write the short-hand electron configurations for the following ions:

   Ni$^{2+}$ ________________________________

   Co$^{2+}$ ________________________________

   Cr$^{3+}$ ________________________________

   Mg$^{2+}$ ________________________________

   Al$^{3+}$ ________________________________

   Ti$^{4+}$ ________________________________

   b. Based on the electron configurations, what is the main difference between these the metal ions that are colored and the metal ions that are colorless?
3) Assuming that the same amount of ore was used to make both unknown solutions, how much more profitable would the copper-rich mine be? Would it be worth paying 30% more for the better ore? Explain and support your conclusion with calculations.

4) You measure a sample of the filtered waste in the Spec-20 at a wavelength of 810 nm and get an absorbance reading of 0.991. Based on the graph of your standard solutions, calculate the copper concentration of the waste jar.

5) Dilution calculation practice:
   a) How many milliliters of a stock solution of 60.0 mg/mL NaCl would you have to use to prepare 0.500 L of a 12.0 mg/mL solution?

   b) If you dilute 20.0 mL of a 60.0 mg/mL stock solution in to a final volume of 1.00 L, what will be the concentration of the diluted solution?