

Spectroscopic Determination of Cu^{2+}

Purpose: You will revisit a previous laboratory procedure and adapt it to a new goal; you will refresh your knowledge of quantitative spectroscopy.

Background: Last semester we exploited Beer's Law, the fact that the concentration of a lightabsorbing solute is proportional to the amount of light it absorbs. We used a generally applicable protocol to determine the Ni^{2+} concentration of an unknown solution in that experiment. This semester we're going to adapt our previous procedure to determining a different unknown concentration, Cu^{2+} in this case. Notice that the instructions below ask you to draft parts of the lab procedure in your lab notebook BEFORE LAB. Your instructor will check that you have done this and are prepared to do the experiment at the start of the lab period. You will work with a partner on this experiment.

Obtain: One unknown copper (II) solution in a 50.00-mL volumetric flask; one 100.0-mL volumetric flask; two 50.00-mL volumetric flasks; one 10.00-mL pipet; one 5.000-mL pipet; one pipet bulb; one pipet rack; seven Spectronic 20 cuvettes; one cuvette rack

Preparing your first solution: Carefully weigh, to the nearest 0.0001 g, about 5 g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (FW 249.68 g/mol). With a wash bottle, quantitatively transfer the solid copper (II) sulfate into a 100.0-mL volumetric flask. Partially fill this volumetric flask with water (50-75 mL), and then swirl until the solid is completely dissolved. Use the wash bottle to fill the volumetric flask to the fill line. This is your copper (II) **stock solution**. Calculate its approximate concentration BEFORE LAB.

Preparing your other solutions: Adapt our previous procedure (the Ni^{2+} determination from last semester) and enter into your lab notebook—BEFORE LAB—your own protocol for preparing the rest of your copper (II) solutions. Plan to make five known solutions by diluting the stock solution of copper (II). Hint: Your unknown solution will have a concentration within the range 0.0500-0.1500 M, so try to match your protocol to this expectation. In addition to the five known solutions, you should also plan to prepare your blank and your unknown solution. You should eventually have seven cuvettes (the special Spectronic 20 tubes) ready to go to the spectrophotometer. You and your partner will need to compare your plans and then decide what you will actually do together in the lab.

Spectrophotometer measurements: Review our previous procedure for measuring the absorbance of the solutions you prepared, including the instructions for operating the Spectronic 20 instrument. We will use the same procedure for this experiment, except that we'll need to adjust the wavelength at which we measure. The figure below shows the absorbance spectra (amount of light absorbed vs. wavelength) of three copper (II) solutions of differing concentration. You will want to use a wavelength at which absorbance changes significantly with analyte concentration, often the highest point on an absorbance peak is best. Look at the figure below and select an appropriate wavelength. Record that wavelength— BEFORE LAB—as part of the new protocol you've entered into your lab notebook for this experiment.

When you make your absorbance measurements in the laboratory you may need to adjust your wavelength if your solutions exhibit too much absorbance, resulting in an error message on the Spectronic 20 instrument. Therefore you should try measuring your most concentrated solution first, and you should be prepared to adjust the wavelength at which you are measuring. Again, look at the figure below and decide how you will respond if your initial wavelength choice doesn't work out (due to excessive absorbance). Add your wavelength adjustment plan to the new protocol you've entered into your lab notebook for this experiment.

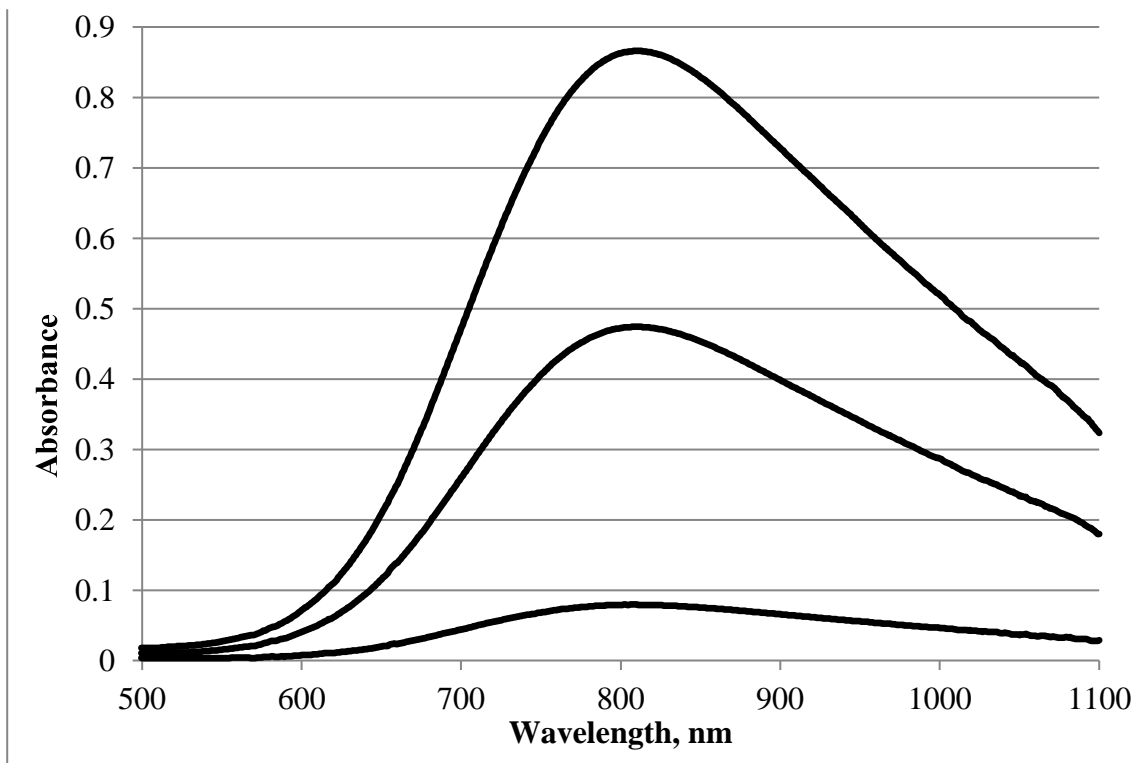


Figure: Visible-to-infrared light absorption spectra of three Cu^{2+} solutions (different concentrations).

Evaluating your data: Once you've measured the absorbances of your six Cu^{2+} solutions (five known and one unknown), you should evaluate your data to determine if you've used an appropriate concentration range for your known solutions. Questions to ask include:

- a) Does the absorbance of the unknown fall near the middle of the range of absorbances covered by the known solutions? (Near the middle would be ideal, but anything within the range of the known solutions will work for this experiment.)

- b) Are some or all of the known solutions too concentrated? (Try to keep absorbance below 1.5)
- c) Are some or all of the known solutions too dilute? (Try to keep absorbance above 0.05)

If your responses to any of these questions reveal potential problems with your data, talk to your instructor about making adjustments to your protocol and repeating the experiment. It is completely normal for scientists to need more than one attempt to work out a new procedure. Maybe consider in advance: If you have to increase or decrease the concentrations of your known solutions, how will you adjust your protocol to do that?

After completing the procedure, but before leaving the lab, write two or three sentences in your laboratory notebook about the quality and reasonableness of the data you collected for this experiment. Note what you might do differently if you had to do this experiment again.

Determining your final result: You should have a very clear idea of exactly what you're going to do with the data you collect in lab in order to determine the Cu^{2+} concentration of your unknown solution. Review last semester's data analysis (for the Ni^{2+} determination) and write your data analysis plan for this lab report in your lab notebook. Again, do this BEFORE LAB. Talk to your instructor during the lab period if you have any questions about the data analysis. Don't leave the lab if you're at all uncertain about how you're going to reach the final result for this experiment: the concentration of your unknown Cu^{2+} solution.