Spectroscopy

In this experiment you will determine the concentration of the Co^{2+} ion and the concentration of the Cu^{2+} in an unknown solution. You will do this by using a spectrometer that measures absorbance of light in the visible range of electromagnetic radiation. You will prepare a standard curve by measuring the absorbance of 6 different known solutions that contain the Co^{2+} ion and the Cu^{2+} ion that you will prepare from $Co(NO_3)_2{\cdot}6H_2O$ and $CuSO_4$. You will also measure the absorbance of Co^{2+} and Cu^{2+} in a reference standard and determine your percent error for each of those two ions in your reference standard.

Stockroom

You will need your unknown and a reference standard.

You will also need two 25 mL burets, 6 small test tubes, one cuvette, two weigh boats, one small plastic funnel, and one 50.00 mL volumetric flask.

Chemicals

You will need enough $Co(NO_3)_2{\cdot}6H_2O$ to prepare a 50.00 mL solution of approximately 0.20 M $Co(NO_3)_2(aq)$ and enough $CuSO_4{\cdot}5H_2O$ to prepare a 50.00 mL solution of approximately 0.12 M $CuSO_4$.

Other equipment

You will need a ring stand with a burette clamp, your wash bottle full of D.I. water, a nitrile glove, and your goggles. You will use a spectrometer (Spectronic 200).

Waste disposal

All cobalt and copper containing waste must go in the cobalt/copper waste containers in the hood. All other solutions may go down the sink drain and all other solid waste may go in the garbage.

Safety

Wear your goggles the entire time. Do not ingest any cobalt or copper containing compounds, they are **TOXIC**. Wear disposable gloves while handling cobalt and copper.

Procedure

Standard solutions

Prior to the beginning of lab calculate the approximate mass of $Co(NO_3)_2 \cdot 6H_2O$ needed to prepare 50.00 mL of approximately 0.20 M $Co(NO_3)_2$ solution. Also calculate the approximate mass of $CuSO_4 \cdot 5H_2O$ needed to prepare 50.00 mL of approximately 0.12 M $CuSO_4$. Do these calculations as part of your pre-lab.

Weigh out approximately the mass of $Co(NO_3)_2 \cdot 6H_2O$ you calculated into a weigh boat that has been tared on the balance. Record the mass of $Co(NO_3)_2 \cdot 6H_2O$ to 3 places past the decimal in your data table.

Weigh out approximately the mass of $CuSO_4 \cdot 5H_2O$ you calculated into another weigh boat that has been tared on the balance. Record the mass of $CuSO_4 \cdot 5H_2O$ to 3 places past the decimal in your data table.

Transfer the $Co(NO_3)_2 \cdot 6H_2O$ and the $CuSO_4 \cdot 5H_2O$ to your 50.00 mL volumetric flask using the plastic funnel. Rinse the beaker and plastic funnel with D.I. water from your wash bottle to ensure that all of the $Co(NO_3)_2 \cdot 6H_2O$ and the $CuSO_4 \cdot 5H_2O$ have been transferred to the volumetric flask.

Add D.I. water to the volumetric flask until it is about half full. Swirl to dissolve all of the $Co(NO_3)_2 \cdot 6H_2O$ and $CuSO_4 \cdot 5H_2O$. Next, add D.I. water until the bottom of the meniscus just touches the top of the 50.00 mL mark on the neck of your volumetric flask. Insert the stopper and invert the flask several times to ensure complete mixing. This is your **stock solution**.

Label your five small test tubes as #1, #2, #3, #4, and #5.

Wash both burettes with D.I. water 3 times. Fill one burette with D.I. water, making sure that you've removed any air bubbles from the tip and that the level is somewhere **beneath** the 0.00 mL mark. Record the initial volume of water in your burette in your data table.

NOTE: THESE BURETTES SHOULD BE READ TO THE NEAREST 0.01 mL!

Rinse the second burette with a few mL of your stock solution. Make sure to collect all waste cobalt and copper solutions in a beaker to put into the cobalt/copper waste container in the hood. Fill the burette with your stock solution. Make sure that you've removed any air bubbles from the tip and that the level is somewhere **beneath** the 0.00 mL mark. Record the initial volume of stock solution in your burette in your data table.

To test tube #1, add about 1 mL of your stock solution from the stock burette. Record the final volume reading on the burette to two places past the decimal. Now add about 4 mL of D.I. water to test tube #1 from the D.I. burette. Record the final burette reading to two places past the decimal.

Follow the same procedure for test tubes #2 - #5, except add about 2 mL of stock and 3 mL of D.I. to test tube #2, 3 mL of stock and 2 mL of D.I. to #3, 4 mL stock and 1 mL D.I. to #4, and 5 mL of stock to #5 (no D.I. to #5). Make sure to record the initial and final burette readings for all additions.

Wearing a glove, put your finger over the top of each test tube and invert it several times to ensure complete mixing.

Finding λ_{max}

Using test tube #1, use the spectrometer (instruction provided by your instructor) to find the wavelength that gives the maximum absorbance for Co^{2+}. This is your λ_{max} for Co^{2+}. Record it in your data table. Read the absorbance and record this as the absorbance for test tube #1 for Co^{2+}.Next, find the wavelength that gives the maximum absorbance for Cu^{2+} with the solution from test tube #1 (this will be at a higher wavelength than that of Co^{2+}). Record this in your data table along with the absorbance.

Beer's Law Plot

Adjust the wavelength of the spectrometer to your λ_{max} for Co^{2+}. Measure the absorbance of test tube #2. Change the wavelength to the one for Cu^{2+} and record the absorbance in your data table. Do the same (wavelength for Co^{2+} and that for Cu^{2+}) for test tubes #3 – #5. Make sure to rinse the cuvette each time with the solution you are going to measure the absorbance of each time. Make sure to go from lowest concentration (test tube #1) to highest concentration (test tube #5). Measure the absorbance of your the reference standard as well as your unknown at the wavelength for Co^{2+} and for wavelength for Cu^{2+} in the cuvette they came in. Record all absorbance measurements in your data table.

Calculations

You will need to calculate:

1.) The approximate mass of $Co(NO_3)_2 \cdot 6H_2O$ needed to make 50.00 mL of approximately 0.30 M Co^{2+} solution as well as the approximate mass of $CuSO_4 \cdot 5H_2O$ needed to make 50.00 mL of approximately 0.12 M Cu^{2+} solution. This must be done as part of the pre-lab.

2.) The $[Co^{2+}]$ and the $[Cu^{2+}]$ in your stock solution as well as every one of the test tubes.

3.) The $[Co^{2+}]$ and the $[Cu^{2+}]$ in the reference standard.

4.) The $[Co^{2+}]$ and the $[Cu^{2+}]$ in your unknown.

  a.) Using your favorite spreadsheet program plot absorbance vs. $[Co^{2+}]$ making an XY scatter plot with the best-fit straight line.

    i.) Include the equation of the best-fit straight line. Make sure to include a title (with your name underneath the title) and to label both axis including units. Cut and paste the graph (not the data) into your lab report.

    ii.) Do the same for the absorbance vs. $[Cu^{2+}]$ graph.

  b.) You will use the equation of the best fit straight line from your Beer's law plot (from your spreadsheet) to calculate the $[Co^{2+}]$ in the reference standard and your unknown as well as the $[Cu^{2+}]$ in the reference standard and your unknown.

    i.) Plug in the absorbance of the reference stand and unknown for "y" into this equation and solve it for "x" which will be the $[Co^{2+}]$ and the $[Cu^{2+}]$ the reference standard and your unknown.

5.) Your percent error for the $[Co^{2+}]$ and the $[Cu^{2+}]$ in the reference standard.

$$\% \text{ Error} = \frac{(\text{Experimental Value} - \text{True Value})}{\text{True Value}} \times 100\%$$

Conclusion

Report the $[Co^{2+}]$ and the $[Cu^{2+}]$ your stock solution, the $[Co^{2+}]$ and the $[Cu^{2+}]$ in the reference standard from your graph, and the $[Co^{2+}]$ and the $[Cu^{2+}]$ your unknown.

Report your percent error for the reference standard for both Co^{2+} and Cu^{2+}.

Determine and analyze one source of potential experimental error. Please see "How to Determine and Analyze a Source of Experimental Error".