Gravimetric Analysis

In this experiment you will determine the concentrations of two ions in an unknown solution. The ions are Cu^{2+} and Pb^{2+}. You will also determine the percent copper in an unknown.

STOCKROOM

You will need two unknowns, bring a large test tube for the Cu^{2+}/Pb^{2+} unknown solution. Matt will give you your solid unknown in a small test tube.

You will need a 10.00 mL volumetric pipette, a hot plate/stirrer, two 100 mL beakers, a magnetic stir bar, a rubber policeman on a glass stir rod, and an extra glass funnel.

CHEMICALS

You will need about 20 mL of 6 M NaOH solution and about 10 mL of 0.4 M Na_2SO_4.

OTHER EQUIPMENT

In addition to the two 100 mL beakers and glass funnel that you received from Matt, you will need the glass funnel from your locker. You will also need your wash bottle full of D.I. water, 2 iron rings, 1 ring stand, 3 pieces of 12.5 cm filter paper, a 250 or 300 mL beaker, a pipette bulb, a watch glass, your 50 mL graduated cylinder, a magnetic stir plate with heat, a magnetic stir bar, and your goggles.

WASTE DISPOSAL

All copper and lead containing waste must go in the copper/lead 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 copper or lead containing compounds, they are **TOXIC**.

PROCEDURE

COPPER UNKNOWN (1st week)

Place your 250 mL beaker on the balance and tare the balance. Add between 1.8 and 2 grams of your solid unknown to the beaker and record the mass to 3 places past the decimal in your data table.

Obtain a piece of 12.5 cm filter paper and label it with your name and what it will have on it (CuO Part A).

Use pencil only to label your filter paper.

Weigh the filter paper and record the mass to three places past the decimal in your data table.

Bring the beaker back to your work station and add about 10 mL of D.I. water to the beaker.

Make sure all of the solid unknown has dissolved by breaking it up with the rubber policeman before adding the NaOH.

Add 10 mL of 6.0 M NaOH solution to the beaker and add your magnetic stir bar to the beaker. Place the beaker on your magnetic stir plate with heat and turn on the stirrer and heat (about half way). **YOU MUST CONTINUOUSLY STIR THE SOLUTION TO AVOID IT SPLATTERING!**

If any of the solid spatters onto the sides of the beaker wash it back down into the solution with D.I. water from your wash bottle. The reaction is complete when the solid has all turned a brown color.

Once the reaction is complete, remove the beaker from the stir plate and place it on your wire gauze on the lab bench.

Fold the pre-weighed piece of filter paper and place it in your glass funnel. Wet it with D.I. water so that it seals against the glass funnel.

Filter the solution in your beaker, collecting the filtrate in a beaker underneath the glass funnel. Make sure to get all of the CuO from the beaker onto the filter paper, using D.I. water to wash it onto the filter paper.

Once the solution has filtered through the filter paper, wash the CuO on the filter paper with D.I. water two times. Discard the filtrate. Place the filter paper with the wet copper (II) oxide in an oven set at 50° C. Leave it there until the next lab period.

UNKNOWN SOLUTION (1st week)

Label two pieces of 12.5 cm filter paper. On one write your name and "$PbSO_4$". On the other write your name and "CuO Part B". Weigh each piece of filter paper individually, recording the masses to three places past the decimal in your data table.

Transfer 10.00 mL of your unknown solution to a 100 mL beaker (beaker A) using a volumetric pipette. Add about 3 mL of 0.4 M sodium sulfate solution (use the transfer pipette next to the bottle). A precipitate should form (the precipitate is lead(II) sulfate).

Place the pre-weighed piece of paper labeled "Pb^{2+}" in a glass funnel in an iron ring on a ring stand. Wet the filter paper with D.I. water from your wash bottle so that it seals against the glass funnel.

Filter the solution in your 100 mL beaker, placing your second 100 mL beaker (beaker B) underneath the funnel to collect the filtrate. The filtrate still contains the Cu^{2+} ion, and maybe some Pb^{2+} ion. Add about 2 mL of 0.4 M sodium sulfate to beaker B. If any precipitate forms filter it through the same filter paper, collecting the filtrate in beaker A. Continue this cycle, alternating between beaker A and beak B to collect the filtrate, until no precipitate forms (this should be no more than 3 or 4 times). Wash the precipitate two times with D.I. water.

Remove the filter paper from the glass funnel and place it in an oven set at 50° C until next lab period.

Add 10 mL of 6.0 M NaOH solution to the beaker and add your magnetic stir bar to the beaker. Place the beaker on your magnetic stir plate with heat and turn on the stirrer and heat (about half way). **YOU MUST CONTINUOUSLY STIR THE SOLUTION TO AVOID IT SPLATTERING!**

If any of the solid spatters onto the sides of the beaker wash it back down into the solution with D.I. water from your wash bottle. The reaction is complete when the solid has all turned a brown color.

Once the reaction is complete, remove the beaker from the stir plate and place it on your wire gauze on the lab bench.

Fold the pre-weighed piece of filter paper labeled CuO Part B and place it in your glass funnel. Wet it with D.I. water so that it seals against the glass funnel.

Filter the solution in your beaker, collecting the filtrate in a beaker underneath the glass funnel. Make sure to get all of the CuO from the beaker onto the filter paper, using D.I. water to wash it onto the filter paper.

Once the solution has filtered through the filter paper, wash the CuO on the filter paper with D.I. water two times. Discard the filtrate. Place the filter paper with the wet copper (II) oxide in an oven set at 50° C. Leave it there until the next lab period.

NEXT LAB PERIOD

Remove the three pieces of filter paper from the oven and weigh each of them. Record the masses to three places past the decimal in your data table.

Calculate the mass percent copper in your copper unknown. Calculate the molarity of Cu^{2+} and Pb^{2+} in your unknown solution.

CALCULATIONS

COPPER UNKNOWN PART A

Mass % Copper in Unknown Part A

$$\frac{\text{Mass of CuO Collected (g)}}{\text{Molar Mass of CuO}} = \text{ mol CuO}$$

$$\text{mol CuO} \times \left(\frac{\text{1 mol Cu}}{\text{1 mol CuO}}\right) = \text{ mol Cu}$$

$$\text{mol Cu} \times (\text{ Molar Mass Cu}) = \text{ mass Cu}$$

$$\frac{\text{Mass Cu}}{\text{Mass of Unknown}} \times 100 = \text{ Mass \% Cu}$$

UNKNOWN SOLUTION PART B

Molarity of Cu^{2+} in Unknown Solution

$$\frac{\text{Mass of CuO Collected (g)}}{\text{Molar Mass of CuO}} = \text{ mol CuO}$$

$$\text{mol CuO} \times \left(\frac{\text{1 mol } Cu^{2+}}{\text{1 mol CuO}}\right) = \text{ mol } Cu^{2+}$$

$$\frac{\text{mol } Cu^{2+}}{0.01000 \text{ L}} = [Cu^{2+}]$$

Molarity of Pb^{2+} in Unknown Solution

$$\frac{\text{Mass of } PbSO_4 \text{ Collected (g)}}{\text{Molar Mass of } PbSO_4} = \text{ mol } PbSO_4$$

$$\text{mol } PbSO_4 \times \left(\frac{\text{1 mol } Pb^{2+}}{\text{1 mol } PbSO_4}\right) = \text{ mol } Pb^{2+}$$

$$\frac{\text{mol } Pb^{2+}}{0.01000 \text{ L}} = [Pb^{2+}]$$

CONCLUSION: For the solid copper unknown report the mass percent copper in the unknown. For the unknown solution report the molarity of Pb^{2+} and the molarity of Cu^{2+} in the solution.

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