A Laboratory Experiment to Review Basic Chemistry Lab Techniques

Apparatus required:

Bench spectrophotometer and tubes
Hot-plate/magnetic stirrer and stirring bar
100/150 mL Beakers
10 mL and 100 mL graduated cylinders
25 mL and 50 mL volumetric flasks
Buchner funnel and filter paper to fit
5 mL Pipet; Buret
Wash bottle with deionized water
Vacuum filter flask
Thermometer
Oven at ~ 130℃

Chemicals Required:

Copper(II) sulfate
Sodium hydroxide
3 M sulfuric acid
Potassium iodide
3% Starch solution
Approx. 0.05 M sodium thiosulfate
Ethanol
Acetone

***Time to complete**: 2 - 2 1/2 hours*

SAFETY PRECAUTIONS: EYE PROTECTION MUST BE WORN IN LABORATORY AT ALL TIMES. USE INSULATED GLOVES WHEN HANDLING HOT GLASSWARE

Introduction

This experiment is designed as a revision lab for upper-level students who are returning to in-class instruction and may have had limited or no hands-on general chemistry lab experience during the 2020/2021 pandemic.

Data collected by using basic chemistry apparatus (electronic balance, hotplate/stirrer, drying oven, pipet, buret, volumetric flasks, bench spectrophotometer) and techniques (inorganic synthesis, filtration, dilution, titration) and will be used to complete important chemistry calculations involving moles, molarity, percent & theoretical yields, limiting & excess reactants, and dilutions. The main reactant, copper(II) sulfate, is regenerated and may be recycled for use in future classes.

Students who have completed the general chemistry lecture sequence (including online) will be familiar with the calculations required to complete this lab. Prior to lab, students may view the accompanying YouTube video describing the entire experiment (link: https://youtu.be/UvbQMwHmz24) which also demonstrates the apparatus and techniques used. Be sure to use YOUR measurements for calculations, not data see in the video.

Background

The reaction of copper(II) sulfate with sodium hydroxide yields insoluble copper(II) hydroxide:

$$CuSO_4 \cdot 5H_2O\ (aq) + 2NaOH\ (aq) \rightarrow Cu(OH)_2\ (s) + Na_2SO_4\ (aq) + 5H_2O\ (l) \qquad \textit{Equation 1}$$

When heated in solution, the insoluble blue hydroxide converts to copper(II) oxide:

$$Cu(OH)_2\ (s) \rightarrow CuO\ (s) + H_2O\ (aq) \qquad \textit{Equation 2}$$

CuO reacts with sulfuric acid to reform aqueous copper(II) sulfate:

$$CuO\ (s) + H_2SO_4\ (aq) \rightarrow CuSO_4\ (aq) + H_2O\ (l) \qquad \textit{Equation 3}$$

The above three reactions are completed during this experiment and the CuO isolated by vacuum filtration, then converting it back to copper(II) sulfate. By measuring the absorbances of the initial and reformed copper(II) sulfate solutions, the percent recovery of the copper can be estimated. The copper content of the reformed solution is also estimated by titration with sodium thiosulfate. Using pipets and volumetric flasks will also provide experience with solution dilutions and calculations.

Procedure

Your instructor will provide details about how to write and submit reports. Record observations and measurements on the data sheet. Use that data to answer the questions at the end of the lab. Reports should include the data sheet and answers to the questions. Show all calculation steps and reasoning for full credit. For calculations, give answers to the correct number of significant figures.

Experimental

Part A: Preparation of $Cu(OH)_2$ and conversion to CuO

Step 1: Weigh about 1.2 g of NaOH pellets into a 100 mL beaker (be sure to keep the lid on the NaOH bottle at all times when not in use). *Record the accurate mass of sodium hydroxide on your data sheet.* Add 10 mL (graduated cylinder) of deionized water. Set aside and swirl occasionally to dissolve the solid while proceeding to step 2.

Step 2: Weigh exactly 3.00 g of $CuSO_4 \cdot 5H_2O$ into a 150 mL beaker (or onto a plastic weighing dish or weighing paper then transfer to a beaker). *Record the mass of copper(II) sulfate on your data sheet.* Add 40 mL of deionized water (graduated cylinder) and a magnetic stirring bar. Stir until the solid dissolves. (The solid does not need heating to dissolve, but the hotplate can be turned on to medium heat at this point for use in step 3). Transfer the copper(II) sulfate solution via a funnel to a 50 mL volumetric flask, rinsing the beaker with a little deionized water (~5 mL) adding the rinsing to the flask. Fill the flask to the mark using the wash bottle and shake to mix.

Step 3: Measure the absorbance of the solution at 650 nm with a bench spectrophotometer, using deionized water as the blank. *Record the absorbance on your data sheet*.

Step 4: Transfer all the copper sulfate solution (from the volumetric flask and spectrophotometer tube) back into the 150 mL beaker, rinsing both the flask and tube with 1-2 mL of deionized water and adding to the beaker. Place the stirring bar back into the solution and place the beaker on the hotplate with rapid stirring and heating. Add the sodium hydroxide solution. A blue precipitate will form.

Step 5: Heat the solution to around 60 ℃ for about 5 min or until all the blue color disappears and a black-brown solid forms. Remove the beaker from the hotplate.

Step 6: Collect the product by vacuum filtration using a Buchner funnel containing a filter paper moistened with a little water from a wash bottle. (When beginning the filtration, first turn on the vacuum so the moist filter paper grips the funnel then pour a little of the dark solution onto the center of the filter paper. Then continue to add more until all has been transferred to the funnel). Rinse any solid from the beaker into the funnel with the wash bottle, keeping the magnetic stirring bar in the beaker. The filtrate should be colorless. As the filtration nears completion and while the precipitate in the funnel is still quite moist, pour 15 mL of water into the funnel to wash the precipitate. When most of the water has washed through, remove the flask from the vacuum and discard the filtrate. Reconnect the vacuum and continue filtering by washing the dark precipitate with ethanol 3 times (3 x 5 mL). Wash an additional two times with 5 mL portions of acetone. Keep the vacuum attached until no more solvent washes through and the solid begins to dry. Discard the ethanol/acetone filtrate into an organic waste bottle.

Step 7: Place the funnel (labeled with your name) with the precipitate in a 130℃ oven for about 10-15 min until the dark solid is dry. (While waiting for the solid to dry in the oven, the reagents and apparatus for the titration in Part C can be collected and assembled, eg buret can be cleaned and filled with sodium thiosulfate solution). Remove the funnel from the oven using insulated gloves and allow to cool to room temperature.

Step 8: Weigh an empty, clean, dry 100 mL beaker. *Record the mass of the empty beaker on your data sheet.* Carefully scrape the dry dark solid from the filter paper into the beaker using a spatula. Any solid adhering to the funnel should also be scraped away and added to the beaker. (If any lumps of the solid still appear wet, the beaker can be placed in the oven for a few more minutes to completely dry, after which it is removed and cooled before weighing). *Record the mass of the beaker plus dry product on your data sheet and determine the mass of dry product, CuO.*

Part B: Conversion of CuO back to copper(II) sulfate

Step 9: Use a wash bottle to rinse (3-4 mL) any CuO left on filter paper or Buchner funnel into the beaker containing the weighed CuO (to ensure complete conversion back to $CuSO_4$). Add 30 mL deionized water to the beaker, a stirring bar, and 5 mL of 3M sulfuric acid (graduated cylinder). Heat the solution at about 50 ℃ with stirring until it turns blue and all the CuO dissolves (~ 5 min). Remove from the hotplate and cool slightly.

Step 10: With a small funnel, transfer the solution to a clean 50 mL volumetric flask (can be wet with water), rinsing out the beaker with a little deionized water and adding to the flask. Fill to mark.

Step 11. Pour some of the copper(II) sulfate solution into a dry spectrophotometer tube (or rinse a wet tube with a little of the solution) and measure its absorbance at the same wavelength used in step 3. Use deionized water as the blank. *Record the absorbance on your data sheet*. Pour the remaining copper(II) sulfate solution from the volumetric flask into a clean 100 mL beaker (add the portion in the spectrophotometer tube to the copper waste bottle provided by the instructor). The solution will be used in Parts C and D.

Part C: Titration of reformed copper(II) sulfate

Step 12: Pipet 5.00 mL of the reformed copper(II) sulfate solution in the beaker from step 11 into a 125 mL Erlenmeyer flask. Add 20 mL deionized water (graduated cylinder) followed by 1.50 g of potassium iodide and swirl to dissolve. A precipitate will form and the solution will turn brown.

Step 13: Titrate the copper solution with approx. 0.05 M sodium thiosulfate solution. *Record the actual molarity on your data shee*t. To perform the titration, first add about 5-10 mL of the sodium thiosulfate from the buret until the darker brown color lightens to an orange-brown. Add 2 mL (graduated cylinder) of 3% starch solution. The solution will turn a dark black-blue color. Continue titrating until one drop turns the solution white (there will also be a white precipitate). *Record the total volume of sodium thiosulfate solution added, including the first addition prior to adding the starch).* Titrations should normally be repeated several times for accuracy and if time permits your instructor may require this. Discard the titrated solution into an appropriate waste container.

Part D: Dilution of reformed copper(II) sulfate

Step 14: Pipet 5.0 mL of the reformed copper(II) sulfate solution from the beaker in step 11 to a 25 mL volumetric flask and dilute with water to the mark. Measure the absorbance of the diluted solution at the same wavelength used in step 3. Use deionized water as the blank. *Record the absorbance of the diluted copper solution on your data sheet.*

Step 15: Place all unused copper(II) sulfate solutions in a class recycle container.

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DATA SHEET

Name _____ Date: _____

Class/Section _____ Instructor _____

Lab Partners (if any) _____

Part A

Mass of sodium hydroxide used: _____

Mass of copper(II) sulfate used: _____

Absorbance of original copper(II) sulfate solution: _____

Mass of beaker plus dry CuO: _____

Mass of empty beaker: _____

Mass of dry CuO: _____

Part B

Absorbance of reformed copper(II) sulfate solution: _____

Part C

Accurate molarity of sodium thiosulfate solution: _____

Total volume of sodium thiosulfate used in titration: _____

Part D

Absorbance of diluted copper(II) sulfate solution: _____

QUESTIONS & CALCULATIONS

Your report should include the completed data sheet and answers to the following questions. Show all calculation steps and reasoning for full credit. Give answers to the correct significant figures.

Q1. Calculate the molar masses of $CuSO_4 \cdot 5H_2O$, NaOH, $Cu(OH)_2$, and CuO.

The balanced equations for the formation of CuO are:

$$\mathbf{CuSO_4 \cdot 5H_2O\ (aq) + 2NaOH\ (aq) \rightarrow Cu(OH)_2\ (s) + Na_2SO_4\ (aq) + 5H_2O\ (l)} \qquad \textit{Equation 1}$$

$$\mathbf{Cu(OH)_2\ (s) \rightarrow CuO\ (s) + H_2O\ (aq)} \qquad \textit{Equation 2}$$

Q2. Determine which of the reactants is limiting and which is in excess in equation 1.

Q3. Calculate the theoretical yield of CuO based on the mass of the limiting reactant.

Q4. Calculate the % yield of CuO. If it is greater than 100%, what does this most likely mean?

Q5. Calculate the molarity of the copper(II) sulfate solution in the original 50 mL volumetric flask.

Q6. According to Beer's Law, $A = \varepsilon bC$, meaning that the concentration (C, molarity) of a solution is directly proportional to its absorbance (A). Provided ε and b remain constant for two solutions and the absorbance and concentration of one solution are known, the concentration of a second solution can be estimated.

Compare the absorbance of the original copper(II) sulfate solution with the absorbance of the reformed solution. What does this tell you about the molarity of the reformed copper(II) solution and hence the recovery of the copper(II) sulfate?

Q7. The determination of copper by titration is a standard volumetric analysis using iodine/thiosulfate and a starch indicator. The equations for the reactions are:

$$2Cu^{2+} + 4I^- \rightarrow 2CuI(s) + I_2$$

$$I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$$

Use the titration data to calculate the molarity of the reformed copper(II) sulfate solution. How does this compare with the molarity calculated in Q5?

Q8. Use $M_1V_1 = M_2V_2$ to determine the concentration of the diluted solution.

Q9. Use Beer's Law to determine the concentration of the diluted solution.

Q10. How do the answers to Q8 and Q9 compare?