REPORT FORM

SYNTHESIS AND ANALYSIS OF A COPPER COMPLEX

Name _____ Section _____ Grade _____/30

The objective of this experiment is to determine the molecular formula of a coordination compound of copper. You know only that its general formula is $Cu(NH_3)_x(SO_4)_y \bullet z\ H_2O$, and you wish to know the values of *x, y* and *z*. To determine this, you determine the mass percentage of each constituent [Cu, NH_3, and SO_4^{2-}] by an appropriate analytical method and then determine the amount of water by difference.

A. Synthesis

Mass of $CuSO_4 \bullet 5\ H_2O$ used in the synthesis. _____ g

Mass of product isolated = _____ g

B. Gravimetric Analysis for Sulfate

To analyze your compound for the mass percent of sulfate, you precipitate the sulfate ion as $PbSO_4$. Knowing the molar mass of $PbSO_4$, and knowing the actual mass of $PbSO_4$ isolated from the sulfate in your compound, you can calculate the mass percent of sulfate in the coordination compound. Consult the experiment done earlier this semester on the analysis of $BaCl_2$ for an outline of the type of calculations done for a gravimetric analysis.

Experimental Data

	Sample 1	Sample 2
Mass of paper + sample		
Mass of paper		
Mass of sample		
Mass of $PbSO_4$ + filter paper		
Mass of filter paper		
Mass of $PbSO_4$		

Calculations

	Sample 1	Sample 2
Moles of $PbSO_4$		
Moles of SO_4^{2-} ion in the $PbSO_4$ sample		
Mass of SO_4^{2-} in $PbSO_4$ precipitate (g)		
Mass % SO_4^{2-} in unknown copper compound		
Average Mass % SO_4^{2-}		

C. Volumetric Analysis for Ammonia

To analyze your compound for the mass percent of NH_3, you titrate a weighed sample with standard HCl. This will give you the number of moles of NH_3 in the weighed sample, a result that will give you in turn the mass of NH_3 and finally its mass percent.

Experimental Data

☞ The concentration of the standardized HCl solution is _____ M

☞ The value is given on the stock bottle of acid in the laboratory. Don't forget to write this down while you are in the laboratory.

	Sample 1	Sample 2	Sample 3 (if needed)
Mass of paper + sample			
Mass of paper			
Mass of sample			
Final buret reading (mL)			
Initial buret reading (mL)			
Amount of HCl added (mL)			

Calculations

	Sample 1	Sample 2	Sample 3 (if needed)
Moles of HCl used in titration			
Moles of NH_3 in sample of copper compound (= moles HCl used)			
Mass of NH_3 in sample of copper compound (g)			
Mass % NH_3 in sample of copper compound			
Average Mass % NH_3			

D. Spectrophotometric Analysis for Copper

The mass percent of copper ion in your compound is determined by spectrophotometry. That is, the greater the amount of Cu^{2+} per gram of compound, the more light a given sample will absorb when dissolved in dilute nitric acid. By first calibrating a spectrophotometer with solutions containing known amounts of Cu^{2+}, you can measure the absorbance of your unknown solutions and then determine the amount of Cu^{2+} therein.

Experimental Data

1. Amount of Compound taken for Analysis

	Sample Tube #5	Sample Tube #6
Mass of paper + sample		
Mass of paper		
Mass of sample of copper compound		

2. Concentration Table for Standard Solutions

☞ Concentration of standard Cu^{2+} solution = _____M

(The value is given on the stock bottle of copper(II) solution in laboratory)

Sample Number	mL standard Cu^{2+}	mL 0.1 M HNO_3	$[Cu^{2+}]$ in dilute solution
1	0.0	10.0	0.0
2	4.0	6.0	
3	7.0	3.0	
4	10.0	0.0	

3. Spectrophotometric Data: Absorbance at 645 nm

Sample No.	Trial 1	Trial 2	Trial 3	Trial 4	Average
1					
2					
3					
4					
5					
6					

4. The Calibration Plot

After determining the average absorbance of each reference standard (solutions 1-4) at 645 nm, plot the average absorbance against the copper concentration on graph paper or using the computer programs available. ***Your calibration plot must be handed in with this laboratory report.***

(See the material on "Spectrophotometry" in Section E for the construction of calibration plots. See also the experiment on analysis for copper, Section E, where you prepared a plot of absorbance versus concentration.)

Slope of the line $= \dfrac{\Delta Absorbance}{\Delta Concentration}$ = _____ Intercept = _____

Calculations

Having constructed the calibration plot for your instrument, you can now use the average absorbance of each unknown solution to determine the concentration of the copper ion in those solutions. From that, you can calculate the mass percent of copper in your samples, and the average mass percent of copper in the unknown compound.

	Sample 5	Sample 6
$[Cu^{2+}]$, mol/L		
Mass of copper (g) = (Cu^{2+} concentration)•(0.01 liters)•(63.54 g/mole)		
Mass % copper		
Average mass % copper		

E. Calculation of the Formula of the Coordination Compound

Based on the mass percentages of Cu^{2+}, NH_3, and SO_4^{2-} that you have found experimentally, calculate the number of moles of each component in a 100 g sample of the unknown compound.

Weight Percent	***Experiment***	***Moles per 100 g***	***Calculated***
Weight % Cu^{2+} = Grams of Cu^{2+} in a 100 g sample of unknown		Moles of Cu^{2+} in 100 g	
Weight % NH_3 = Grams of NH_3 in a 100 g sample of unknown		Moles of NH_3 in 100 g	
Weight % SO_4^{2-} = Grams of SO_4^{2-} in a 100 g sample of unknown		Moles of SO_4^{2-} in 100 g	
Total mass of Cu^{2+}, NH_3, and SO_4^{2-}			
Grams of H_2O = 100 - (total of Cu, NH_3, and SO_4^{2-})		Moles of H_2O in 100 g	

In the last column of the table above, you have listed the components of the unknown compound in terms of the number of moles of each in a 100 g sample. You can now determine the formula of the compound by taking the ratio of the number of moles of each substance (NH_3, SO_4^{2-}, H_2O) to the number of moles of Cu^{2+}.

Moles of NH_3 per moles Cu^{2+} _____

Moles of SO_4^{2-} per moles of Cu^{2+}

Moles H_2O per moles Cu^{2+}

Rounding the ratios above to the nearest whole number, fill in the blanks below to give the empirical formula of the copper coordination compound.

$Cu(NH_3)_x(SO_4)_y \bullet z\ H_2O$ x = ____ y = ____ z = ____

F. Calculating the Percent Yield

NOTE: These calculations can only be done *after* you have completed all the experimental work and have done the calculations on the last page of this form.

1. Mass of $CuSO_4 \bullet 5\ H_2O$ used in the synthesis (from the first page of this Report Form _____g

2. Moles of $CuSO_4 \bullet 5\ H_2O$ used in the synthesis (M = 249.7) _____moles

3. Grams of product isolated _____ g (from first page of this Report Form)

4. Molecular formula you have determined _____

5. If each mole of $CuSO_4 \bullet 5\ H_2O$ used in the synthesis leads to one mole of product, how many moles of product do you expect? _____

6. What is the theoretical yield of product? _____ g

7. Calculate the percentage yield based on your formula. _____ %