Analysis of Ni Complex Lab Worksheet

By reviewing the material in sections 3.1-3.5 in your text, and reading the lab, you should be able to get a handle on these calculations. Although the compound used for these calculations is fictional, every calculation that you perform is identical to those required in the lab. Since each week of this lab is worth 20 points, it is worth investing the time to figure out what you need to do prior to the lab. You will be permitted to use your own set of calculations for this worksheet as a reference when performing the lab write-up; you will not be permitted to use another student's work. Note that the WEEK 2 calculations are more involved than WEEK 1.

WEEK 1 Practice Sheet

You are given 5.0 g of a hypothetical compound $A_wB_xC_yD_z$ in some water. Upon dissolving, the compound splits apart into A(*aq*), B(aq), C(*aq*) and D(*aq*). B is a base that undergoes the following reaction with HCl:

$$B + 2\,HCl \rightarrow H_2B^{2+} + 2Cl^-$$
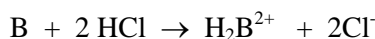
In order to determine the number of moles of B in the compound you perform a titration. First you dissolve 0.500 grams of the compound in 20 mL of water. You then perform the titration and find that it takes 20.00 mL of 0.0100 M HCl to reach the end point of the titration. The molar mass of B is 500.0 g/mol.

Based on the information from above, complete the following:

Moles of HCl used in titration: _____ (Answer: 2.00×10^{-4} mol)

Moles of B titrated: _____ (Answer: 1.00×10^{-4} mol)

Mass of B titrated:_____ (Answer: 5.00×10^{-2} g B)

Mass % of B in the compound:_____(Answer: 10.0% B)

Moles of B in 100 grams of the compound: _____ (Answer: 0.0200 mole)

WEEK 2 Practice Sheet

Reminder: There are 0.0200 moles of B in 100 grams of the compound: $A_wB_xC_yD_z$ (Week 1).

You dissolve 0.250 g of your compound ($A_wB_xC_yD_z$) in exactly 10.00 mL of water producing 10.00 mL of solution. Using the photometer and standard solutions, you find that the concentration of A is 2.00 x 10^{-2} M. The molar mass of A is 750.0 g/mol.

Moles of A in the solution sample:_____(Answer: 2.00 x 10^{-4} mol A)

Mass of A in the solution sample: _____ (Answer: 0.150 g A)

Mass % of A in the compound: _____(Answer: 60.0% A)

Moles of A in 100 grams of the compound: _____ (Answer: 0.0800 mol A)

The compound, $A_wB_xC_yD_z$ has an oxidation number of 0 (zero). The oxidation number of A is +1, the oxidation number of B is +2, the oxidation number of C is –3, and the oxidation number of D is 0. The molar mass of C is 500.0 g/mol.

Moles of A in 100 grams of the compound: 0.0800 mol A

Moles of B in 100 grams of the compound: 0.0200 mol B

Moles of C in 100 grams of the compound (use oxidation numbers): _____   (Answer: 0.0400 mol C)

Grams of C in 100 grams of the compound:_____ (Answer: 20.0 g C)

Mass % of C in the compound:_____ (Answer: 20.0% C)

The molar mass of D is 166.7 g/mol.

Mass % D in the compound:_____ (Answer: 10.0%)

Mass of D in 100 grams of the compound:_____ (Answer: 10.0 g D)

Moles of D in 100 grams of the compound:_____ (Answer: 0.0600 mol D)

Formula of compound: _____ (Answer: $A_4BC_2D_3$)

ANALYSIS OF A NICKEL COMPLEX

**INTRODUCTION**

This lab will span the next two lab sessions. Throughout both weeks you will be analyzing a coordination complex (a compound) containing nickel. Coordination complexes are an important class of compounds. They are found in paint pigments, colored glass, and in both natural and synthetic gemstones. Some complexes perform crucial biological functions.

Coordination complexes consist of a metal ion that is attached (bonded) to elements and/or other compounds called ligands. In this lab the ligands that will be attached to the nickel ion are: H_2O, SO_4^{2-}, and ethylenediamine (abbreviated "en"). Ethylenediamine is a weak base, with the formula $H_2N\text{-}CH_2\text{-}CH_2\text{-}NH_2$. For background on coordination complexes, you may want to consult Section 22.3 in your text.

The complex that you will analyze was prepared by reacting ethelyenediamine, $NiSO_4$ and water, forming a product with general formula:

$$[Ni_v(en)_w (H_2O)_x (SO_4)_y] \cdot z\ H_2O$$
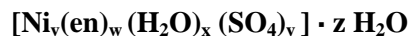
The amount of the reactants determines how many of each ligand will attach to the nickel ion and hence the color and makeup (the values of v, w, x, y and z) of the product. Following the procedure outlined below, you will analyze a compound of unknown composition; i.e. you will not know the values of v, w, x, y, and z. Through careful work using a variety of techniques that you have learned this semester, you will be able to determine a likely chemical formula of the product.

During the first week you will determine by titration, the mass percentage of ethylenediamine in your compound, which will then allow you to calculate:

w= moles of ethylenediamine in the compound

The procedure for the second week will outline how you can determine/calculate:

v = moles of Ni^{2+}
y = moles of SO_4^{2-} in the compound
$x+ z$ =moles of H_2O in the compound (note that you only will know the sum of $x+ z$)

Once you have all of this information, you should be able to determine the formula of the complex.

In this lab you will have to perform several calculations of mass percentage. Be sure to review Sections 3.1-3.5 of your text.
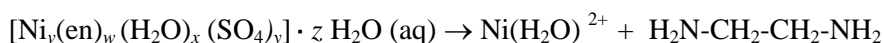
PROCEDURE (Week 1)

Titration of ethylenediamine with standard HCl solution
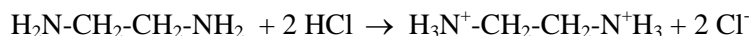
This first procedure is to determine the number of ethylenediamine (H_2N-CH_2-CH_2-NH_2) molecules attached to the nickel ion(s) in your compound. Be sure to pay attention to the stoichiometry of the titration reaction!

1. Green aqueous Ni^{2+} ion is released into the solution when the complex dissociates. This masks the endpoint of the indicator. To account for the color masking, prepare an artificial endpoint solution. Add 5 drops of the indicator to a titration flask with 0.1 g of $NiSO_4 \cdot 6\,H_2O$ dissolved in 20 mL of the standardized acid. Use the color of this solution as your permanent end point indicator when performing the titration. If the color fades while performing the titration, continue adding HCl.

2. Weigh a sample (about 0.15 g) of the assigned complex and add it to an Erlenmeyer flask (be sure to record the exact mass). Add 10 mL of water and completely dissolve your sample. After the entire sample has dissolved, add 5 drops of indicator, bromcresol green, and titrate with a standardized acid to the <u>yellow</u> end point. Repeat the titration at least twice or until you have consistent data.

3. Your compound dissociates **very slowly** in water according to the equation below; therefore be patient enough to ensure that the sample is completely dissolved before titrating to the **permanent, lasting end-point**.

    Dissociation Equation

    $$[Ni_y(en)_w\,(H_2O)_x\,(SO_4)_y] \cdot z\,H_2O\,(aq) \rightarrow Ni(H_2O)^{2+} + H_2N\text{-}CH_2\text{-}CH_2\text{-}NH_2$$

    Titration Equation

    $$H_2N\text{-}CH_2\text{-}CH_2\text{-}NH_2 + 2\,HCl \rightarrow H_3N^+\text{-}CH_2\text{-}CH_2\text{-}N^+H_3 + 2\,Cl^-$$

PROCEDURE (Week 2)

Colorimetric Analysis for Ni $^{2+}$

This procedure is used to determine the number of moles of nickel ions in your compound.

1. Double click the "Logger Pro" icon and allow the screen to open.

2. The spectrometer needs to be powered for about 5 minutes before using so do this step before preparing your solutions. Do not use the Go!Link with the spectrometer. Plug the spectrometer via provided USB cable to the computer USB port.

3. Load a 25 mL buret with about 15 mL of $NiSO_4 \cdot 6\ H_2O$ stock solution (**be sure to record the molarity of the stock solution in the lab report**). Load another buret with deionized water.

4. Do not mix the solutions together yet. First, use the given molar concentration of the $NiSO_4 \cdot 6\ H_2O$ stock solution to determine the molar concentration of Ni^{2+} that will be initially in each test tube given in the Table below once <u>all</u> reagents have been mixed together.

5. Clean and label five test tubes as shown in the Table below. Deliver the volumes of nickel stock solution and water shown in the Table. **Do not add the ethylenediamine at this time. Just prior to making each absorbance reading, add 5.0 mL of 10 % ethylenediamine from a pre-set pump dispenser to the test tubes and mix**.

Tube	mL Ni $^{2+}$ stock solution	mL deionized H_2O	mL en	Molar Concentration of Ni^{2+} in solution
A	0	5.0	5.0	
B	1.0	4.0	5.0	
C	2.0	3.0	5.0	
D	3.0	2.0	5.0	
E	4.0	1.0	5.0	

6. Calibrate the Spectrometer ; Do not unplug the spectrometer during this experiment or you will have to start over.

    a. Return to the Logger Pro screen on the computer. Click: Experiment ; Calibrate ; Spectrometer:1.

    b. Allow the lamp to warm up for 90 seconds as displayed on the computer screen.

    c. Add the ethylenediamine to the contents of test tube A (the blank).   Only touch the ridged faces of the cuvette, never touch the clear faces. Rinse and fill a cuvette (about ¾ full) with the blank. **Gently blot** (don't scratch the sides of the cuvette) off any drips on the outside of the cuvette with a Kimwipe.

    d. Place the cuvette containing the blank in the spectrometer so that one of the clear sides is aligned with the white arrow at the top of the cuvette slot. Click: "Finish Calibration" ; OK. Remove the cuvette containing the blank from the spectrometer but keep it handy.

7. Determine the Wavelength of Maximum Absorbance

    a. Add the ethylenediamine to contents of test tube B and mix. Rinse and fill a cuvette with the solution. Place the cuvette in the spectrometer. Click: Experiment ; Data Collection ; Full Spectrum ; Done. Click the rainbow icon labeled "Absorbance=…" in the upper left hand corner of the window. Change the "Wavelength Range" to 350-780 nm. Close this box by clicking the "x" in the upper right hand corner of the window.

    b. Click the small green triangle in the toolbar labeled "Collect". After the line graph appears on the screen, click the small red square in the toolbar labeled "Stop".

    c. To automatically store the maximum wavelength go to the toolbar and select: Experiment ; Store Latest Run.

    d. Immediately do steps 8a through 8c.

8. Generate the Calibration Curve.

    a. In the toolbar click: Experiment ; Data Collection ; choose Events with Entry in the Mode box. Highlight the word Event in the Column Name box and replace it with Concentration. Put M in the Units box. Clear "Short Name". OK. You are ready to begin collecting data. Remove the cuvette from the spectrometer but keep it handy.

    b. Place the cuvette containing the blank (kept from 6d) back in the spectrometer. Click the begin data collection button (triangle) in the toolbar labeled "Collect". When the absorbance reading stabilizes, click the KEEP button (be careful that you **don't** accidentally click the stop data collection button (square)) located in the toolbar just to the right of the Red Stop Button. Type in the concentration of the solution that is in the cuvette (don't include units). OK.

    c. Place the cuvette containing the most dilute solution (kept from 8a) back in the spectrometer. When the absorbance reading stabilizes, click the KEEP button (be careful that you **don't** accidentally click the stop data collection button (square)), and enter the concentration of the analyzed solution.

    d. Working, one solution at a time, in order of most dilute to most concentrated of the remaining standard solutions (contents of test tubes C, D, and E), add the ethylenediamine to the test tube containing the solution and mix. Rinse and then fill a cuvette with the solution that will be analyzed. Place the cuvette in the spectrometer. When the absorbance reading stabilizes, click the KEEP button (be careful that you **don't** accidentally click the stop data collection button (square)), and enter the concentration of the analyzed solution. Repeat until the absorbance of each standard solution has been determined.

        Pour your samples back into the appropriate TT after you have measured each absorbance and discard them in the waste container.

    d. When the absorbance of all standard solutions has been measured, click the stop data collection button (square) located in the toolbar.

    e. To determine the equation of the line for your calibration curve click: Analyze ; Linear Fit. A box should appear with the equation and a correlation.

To receive full credit for this lab your calibration curve must be a good, straight-line graph, with a correlation coefficient of 99% or better (Corr: on the screen reads 0.9900 or greater). You should repeat the experiment until you get this proficiency. Work carefully. If you need to repeat the experiment, you still must be done with the write up and post-lab questions before the end of the lab period.

9. Prepare a solution sample of your Unknown. Weigh between 100 to 150 mg of your compound into a 10 mL volumetric flask (record the mass exactly). Add 4 mL of water to dissolve it. If necessary add 3 M HCl one drop at a time to help your sample dissolve. Once dissolved, add 5.0 mL of 10% ethylenediamine to the 10 mL volumetric flask. Dilute to the 10 mL volume mark with water. Mix thoroughly. To make filling the cuvette easier, pour about 5 mL of this solution into a large test tube. Rinse and fill a cuvette with the solution containing assigned unknown. Place the cuvette in the spectrometer. Record the absorbance in your lab report once the reading has stabilized.

10. Each lab partner's report must have a Logger Pro generated printout of the calibration curve attached to it. The printout must show the graph, the information needed to generate the equation (slope and intercept) for the line, and the correlation reading. To do this click: File ; Print. Uncheck the "Print Visible Spectrum on Wavelength Graphs" and change the "orientation" to landscape under properties. Be sure that the names of all lab partners are entered in the "Name" section and that the date box is checked.

11. When you are done, return your bin to Lab Services.

Determination of sulfate content of your compound

In order to determine the number of sulfate ligands attached to the nickel ion, you need to consider the following information.

- The overall oxidation number on the complex, $[Ni_v(en)_w(H_2O)_x(SO_4)_y] \cdot z\ H_2O$, is zero.
- The oxidation number of water is zero.
- The oxidation number of the sulfate ion is -2.
- The oxidation number of ethylenediamine, en, is zero
- The oxidation number of nickel in the complex is +2.
- The % en and % Ni^{2+} may be used to calculate the number of moles of en and Ni^{2+} present in 100 grams of the compound.

You then should be able to calculate the number of moles of sulfate in 100 grams of the compound.

Water Determination

At this point, you know the mass percentages of all of the ligands except for the water. Keep in mind that the percentages must add up to 100% for the compound. Use this fact to first determine the percentage of water in the compound.

Empirical Formula Determination

The number of moles of each component (i.e., ions, ligands) in 100 grams of the compound can be determined using mass percentages and molar masses.

At this point we will deviate a bit from the method used in the textbook to determine empirical formulas of compounds. Because the sample may include moisture from the air as well as water from the actual compound, the mole ratio of water in the compound to the other components in the compound may not be a whole number.

Instead of dividing the moles of all components by the smallest number of moles, divide the moles of all components (including water) by the smallest of the number of moles of the remaining components (nickel, sulfate, ethylenediamine). If you need to multiply by a number to get the smallest set of whole number subscripts on nickel, sulfate, and ethylenediamine, do so. Multiply the subscript on water by the same number, but don't worry if it does not result in a whole number.

DATA AND ANALYSIS SHEET: ANALYSIS OF A NICKEL COMPLEX: WEEK 1

Name: _____

Date _____   Lab Partner _____

Unknown Identification Number (place this number on the report for week 2 also: _____

Ethylenediamine Titration. Include your work for all results that require calculations.

Molar mass of ethylenediamine: _____

Molarity of standard HCl   _____

	Trial #1	Trial #2	Trial #3
Mass of sample	_____	_____	_____
Initial buret reading	_____	_____	_____
Final buret reading	_____	_____	_____
Total volume acid used	_____	_____	_____
Moles of HCl used	_____	_____	_____
Moles of ethylenediamine titrated	_____	_____	_____
Mass of ethylenediamine titrated	_____	_____	_____
Mass percentage ethylenediamine in the compound	_____	_____	_____

Average mass percentage of ethylenediamine in the compound _____ (**Write in this value on the appropriate blank in the lab report for Week 2**.)

Moles of ethylenediamine in 100 grams of the compound _____ (**Write in this value on the appropriate blanks in the lab report for Week 2**.)

TURN IN THIS PAGE AT THE END OF THE FIRST WEEK OF LAB.

DATA AND ANALYSIS SHEET: ANALYSIS OF A NICKEL COMPLEX: WEEK 2

Name: _____

Date _____   Lab Partner _____

Unknown Identification Number: _____

Include your work for all results that require calculations.

Colorimetric determination of nickel

Molar concentration of $NiSO_4 \cdot 6\,H_2O$ stock solution   _____

Equation of the line for calibration curve   _____

Correlation   _____

Mass of compound used in solution sample   _____

Absorbance of solution sample   _____

Concentration of nickel in the solution sample   _____

Moles of nickel in the solution sample   _____

Mass of nickel in the solution sample   _____

Mass percentage of nickel in the compound   _____

Moles of nickel in 100 grams of the compound   _____

CALCULATIONS FOR LINES MARKED WITH AN * HAVE ALREADY BEEN DONE, JUST TRANSFER THE RESULTS.

Sulfate Content

*Moles of ethylenediamine in 100 grams of the compound (**Week 1**)   _____

*Moles of nickel in 100 grams of the compound   _____

Moles of sulfate in 100 grams of the compound   _____

Mass of sulfate in 100 grams of the compound   _____

Mass percentage of sulfate in 100 grams of the compound   _____

Water Determination

*Mass percentage of ethylenediamine in the compound (**Week 1**)   _____

*Mass percentage of nickel in the compound   _____

*Mass percentage of sulfate in the compound   _____

Mass percentage of water in compound   _____

Moles of H_2O in 100 grams of compound   _____

Empirical Formula Determination

*Moles of ethylenediamine in 100 grams of the compound (**Week1**)   _____

*Moles of nickel in 100 grams of the compound   _____

*Moles of sulfate in 100 grams of the compound   _____

*Moles of water in 100 grams of the compound   _____

Determine the empirical formula of the compound

Empirical Formula of the Compound (Complex)   _____