Name:_____ Section_____

Chemistry 104 Laboratory
University of Massachusetts Boston

Hexamminenickel(II) Chloride Analysis

PRELAB ASSIGNMENT

Calculate the percent ammonia in pure $[Ni(NH_3)_6]Cl_2$. Show your work.

Answer: _____

The purpose of this laboratory is to determine the amount of ammonia that is complexed in the hexamminenickel(II) chloride that you synthesized in the last experiment. The procedure is to add a known quantity of HCl(*aq*) by pipet to a sample of your hexamminenickel(II) chloride salt. The amount of HCl(*aq*) added is more than what is necessary to neutralize all of the NH_3 in the sample. Part of the added HCl(*aq*) reacts with the NH_3 in the hexamminenickel(II) complex according to

$$\underset{\text{in the salt}}{NH_3(\textit{in complex})} + \underset{\text{by pipet}}{HCl(aq)} \rightarrow NH_4^+(aq) + Cl^-(aq) \qquad (1)$$

but the excess HCl(*aq*) remains in solution. The amount of this excess HCl(*aq*) is found by titration; i.e., by reaction with a standardized NaOH(*aq*) solution delivered from a buret according to

$$\underset{\text{excess}}{HCl(aq)} + \underset{\text{by buret}}{NaOH(aq)} \rightarrow H_2O(l) + NaCl(aq) \qquad (2)$$

Thus, the total moles of HCl(*aq*) added by pipet equals the moles of NH_3 in the sample of the complex plus the moles of NaOH(*aq*) delivered at the titration endpoint. This fact is used to calculate the amount of NH_3 as follows:

V_a is the milliliter volume of C_a molar HCl(*aq*) added by pipet. This volume contains V_aC_a millimoles of HCl.

V_b is the milliliter volume of C_b molar NaOH(*aq*) added at the endpoint. This volume contains V_bC_b millimoles of NaOH.

$$\underset{\text{in the salt}}{\text{millimoles of } NH_3} = \underset{(V_aC_a)}{\text{millimoles HCl}} - \underset{(V_bC_b)}{\text{millimoles NaOH}} \qquad (3)$$

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IN THE LABORATORY

While waiting for a balance in Step 1, you may skip ahead to Step 5.

1) Weigh out three samples of hexamminenickel(II) chloride into weighing dishes labeled A, B, and C. The samples should weigh anywhere from 0.09 to 0.14 grams. Record each mass to a tenth of a milligram.

2) Quantitatively transfer (no loss) each of these samples to 200- or 250-mL Erlenmeyer flasks labeled A, B, and C. Add deionized water up to the 50-mL line on each flask.

3) Label a dry beaker "HCl" (Why dry?) and obtain about 100 mL of standardized HCl(*aq*) solution. Rinse a 25-mL pipet with this solution, and then transfer 25 mL of standardized HCl(*aq*) to each flask. Record the concentration C_a of the HCl(*aq*) solution on your data sheet.

4) Add 3 or 4 drops of bromocresol green indicator to each flask. The solution should now be a pale yellow color.

5) Label a *dry* beaker "NaOH" and obtain about 100 mL of standardized NaOH(*aq*) solution. Use a small amount of this solution to rinse a 50-mL buret (including the tip). Then fill the buret with this solution so that the meniscus is anywhere between the 0- and the 3-mL markings. Be sure that there is no air bubble in the tip. Ask your instructor to check your buret.

   Record the concentration C_b of the NaOH(*aq*).

6) Record the initial buret reading to two decimal places; i.e., estimate to a hundredth of a mL. Titrate solution A very slowly and with continuous swirling. The indicator turns from yellow in acid solution to blue in basic solution. The endpoint is indicated by the first *permanent* blue or greenish blue color. It is a very sharp endpoint, changing color within as little as half a drop. Record the buret reading at the endpoint to two decimal places.

   Calculate the endpoint volume V_b as the difference between the endpoint buret reading and the initial buret reading.

7) Refill the buret with NaOH(*aq*) solution if you predict the next titration will go past the 50-mL line. Titrate solutions B and C in the same manner. Be certain to record the initial readings.

8) Calculate the millimoles of NaOH added to each flask at the endpoint. This is also the millimoles of excess HCl, the amount that remained after some reacted with NH_3 in the complex according to reaction (1).

9) Calculate the millimoles of NH_3 reacted with HCl according to reaction (3).

10) Calculate the mass of NH_3 (molecular weight 17.03 g/mol) in the complex salt in each flask.

11) Calculate the percent by mass of NH_3 in the three samples.

12) Calculate the mean and standard deviation of the three results in Step 11.

13) Calculate the percent of NH_3 in pure $[Ni(NH_3)_6]Cl_2$ using this molecular formula and atomic weights.

Hand in your completed Data Sheet and Report page to the instructor before your leave.

Hexamminenickel(II) Chloride Analysis

Name:_____ Section_____

Partner's name: _____

AMMONIA DATA SHEET AND REPORT

Sample	A	B	C
1. Mass of solid	_____	_____	_____
3. Molarity of HCl added by 25.00-mL pipet		C_a = _____	
Millimoles of HCl added by V_a = 25.00-mL pipet		V_aC_a = _____	
5. Molarity of NaOH in the buret		C_b = _____	
6. and 7. Titrations	A	B	C
Initial reading	_____	_____	_____
Final reading	_____	_____	_____
Endpoint volumes, V_b	_____	_____	_____
8. mmoles of NaOH in V_b	_____	_____	_____
9. mmoles NH_3 reacted	_____	_____	_____
10. Mass of NH_3 reacted	_____	_____	_____
11. Percent NH_3 by mass	_____	_____	_____
12. Mean percent NH_3		_____	
Standard deviation of percent NH_3 by mass		_____	
13. Theoretical percent NH_3 by mass		_____	