

Name: \_\_\_\_\_ Section \_\_\_\_\_

Chemistry 104 Laboratory  
University of Massachusetts Boston

## Hexamminenickel(II) Chloride Analysis

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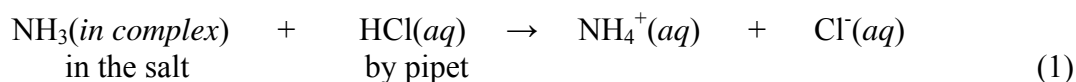
### PRELAB ASSIGNMENT

Calculate the percent ammonia in pure  $[\text{Ni}(\text{NH}_3)_6]\text{Cl}_2$ . Show your work.

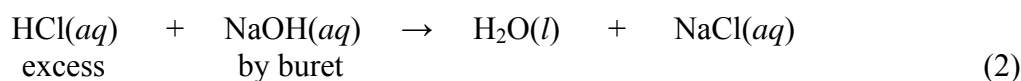
Answer: \_\_\_\_\_

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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  $\text{HCl}(aq)$  by pipet to a sample of your hexamminenickel(II) chloride salt. The amount of  $\text{HCl}(aq)$  added is more than what is necessary to neutralize all of the  $\text{NH}_3$  in the sample. Part of the added  $\text{HCl}(aq)$  reacts with the  $\text{NH}_3$  in the hexamminenickel(II) complex according to



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



Thus, the total moles of  $\text{HCl}(aq)$  added by pipet equals the moles of  $\text{NH}_3$  in the sample of the complex plus the moles of  $\text{NaOH}(aq)$  delivered at the titration endpoint. This fact is used to calculate the amount of  $\text{NH}_3$  as follows:

$V_a$  is the milliliter volume of  $C_a$  molar  $\text{HCl}(aq)$  added by pipet. This volume contains  $V_a C_a$  millimoles of  $\text{HCl}$ .

$V_b$  is the milliliter volume of  $C_b$  molar  $\text{NaOH}(aq)$  added at the endpoint. This volume contains  $V_b C_b$  millimoles of  $\text{NaOH}$ .

$$\begin{array}{ccccccc} \text{millimoles of NH}_3 & = & \text{millimoles HCl} & - & \text{millimoles NaOH} \\ \text{in the salt} & & (V_a C_a) & & (V_b C_b) \end{array} \quad (3)$$

## Hexamminenickel(II) Chloride Analysis

### 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  $\text{NH}_3$  in the complex according to reaction (1).

### Hexamminenickel(II) Chloride Analysis

- 9) Calculate the millimoles of  $\text{NH}_3$  reacted with  $\text{HCl}$  according to reaction (3).
- 10) Calculate the mass of  $\text{NH}_3$  (molecular weight 17.03 g/mol) in the complex salt in each flask.
- 11) Calculate the percent by mass of  $\text{NH}_3$  in the three samples.
- 12) Calculate the mean and standard deviation of the three results in Step 11.
- 13) Calculate the percent of  $\text{NH}_3$  in pure  $[\text{Ni}(\text{NH}_3)_6]\text{Cl}_2$  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		$V_a C_a =$ _____	
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		_____	