Titrimetric Analysis of a Mixture of Sodium Hydrogen Carbonate and Sodium Chloride

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**Objective:** To analyze a mixture of sodium hydrogen carbonate and sodium chloride by an indirect titrimetric procedure.

**Background Information:**

In the exercise SUSB-054, you analyzed a mixture of sodium hydrogen carbonate and sodium chloride by two analytical methods, gasometric and gravimetric. In this exercise you will analyze such a mixture by an indirect titrimetric method, also known as Back-Titration, as follows:

A known mass of the unknown mixture will be reacted with a known amount of hydrochloric acid of known concentration in sufficient excess to neutralize all the sodium hydrogen carbonate in the mixture as shown:

\[
\text{H}^+ (\text{aq}) + \text{NaHCO}_3(\text{s}) \rightarrow \text{Na}^+ (\text{aq}) + \text{H}_2\text{O} (\text{l}) + \text{CO}_2 (\text{g})
\]  

Since an excess is used, some of the hydrochloric acid will remain un-reacted. The un-reacted hydrochloric acid will be determined by titration with a standard sodium hydroxide solution. The amount of acid reacted with sodium hydrogen carbonate will be the difference in the amount of hydrochloric acid initially added and the amount of un-reacted hydrochloric acid and can be related to amount of sodium hydrogen carbonate according to the stoichiometry of reaction (1). This method of determining the quantity of a sought substance indirectly is also called Back-Titration.

The carbon dioxide produced in the reaction is soluble in water by virtue of the following equilibrium:

\[
\text{CO}_2 (\text{g}) + \text{H}_2\text{O} (\text{l}) \leftrightarrow \text{HCO}_3^- (\text{aq}) + \text{H}^+ (\text{aq}),
\]

If carbon dioxide is left dissolved, the \(\text{H}^+\) produced by this equilibrium will also be titrated with sodium hydroxide and the volume of sodium hydroxide recorded at equivalence point will be higher than actual. Therefore, the \(\text{CO}_2\) must be removed. This is accomplished by boiling to shift the above equilibrium to the left. This is also the reason that sodium bicarbonate can not be determined precisely by direct titration with hydrochloric acid.

Since, after removing the \(\text{CO}_2\), the titration is between a strong acid and strong base, the pH at the equivalence point is 7. Bromthymol blue, which has a color change from yellow to blue at pH 7 – 8 range, is a suitable indicator for the titration end point.
**Procedure:**

**Caution!** Sodium hydroxide as well as hydrochloric acid, are corrosive to the skin and **must be washed off immediately in case of accidental contact.**

Obtain two burets from your TA. Fill one buret with standard NaOH solution and the other with standard hydrochloric acid. Don’t forget to rinse the buret with the solution to be filled. Also make sure buret tips have no air bubble trapped. Drain both burets to slightly below zero mark. Record concentration of acid and base solutions indicated on the bottles in your lab notebook. Label each buret to avoid confusion.

Weigh about 0.1 -0.2 gram of unknown mixture in an Erlenmeyer flask by the method of **weighing by difference.** Record the mass to the nearest 0.0002 g in the notebook. Add 20 mL of distilled water into the flask to dissolve solid mixture.

Record the initial reading on both burets to 0.02 mL precision in your notebook. Drain about 30 mL of hydrochloric acid from the buret into the Erlenmeyer flask containing solution of mixture. Heat the Erlenmeyer flask on a hot plate to boiling and allow it to boil for one minute to insure that dissolved carbon dioxide is expelled. Wash the sides of the flask by squirting distilled water from a squeeze bottle. Cool the flask to room temperature.

Add 2 – 3 drops of bromthymol blue indicator. The color of the solution should now be yellow. Titrate the solution with standard sodium hydroxide solution in the second buret. The end point is reached when the color changes from yellow to pale blue by the addition of one final drop of sodium hydroxide and remains blue. If you overshoot the end point and get a deep blue color, back titrate with hydrochloric acid from the first buret dropwise till you see the desired pale blue color after one drop. You may go back and forth at the end point by adding additional drops of acid or base until you see the proper color change.

Record the final reading on both burets. Repeat the procedure till you have three trials within 1% error.

**Calculations:**

According to the stoichiometry of the reaction (Eq 1)

\[ \text{mmoles of NaHCO}_3 \text{ in the mixture} = \text{mmoles of HCl reacted} \]

\[ \text{mmoles of HCl reacted} = \text{mmoles of HCl initially added} - \text{mmoles of HCl remaining} \]

\[ \text{mmoles of HCl initially added} = \text{Molarity of HCl} \times \text{Volume of HCl (mL)} \]

\[ \text{mmoles of HCl remaining} = \text{mmoles of NaOH titrated} = \text{Molarity of NaOH} \times \text{Volume of NaOH} \]

\[ \text{Mass of NaHCO}_3 \text{ in the mixture} = \text{mmoles of NaHCO}_3 \times 84.10 \text{ mg/mmmole} \]

\[ \text{Mass of NaHCO}_3 \text{ (mg)} \times 100 \]

\[ \% \text{ of NaHCO}_3 \text{ in the mixture} = \frac{\text{Mass of NaHCO}_3 \text{ (mg)}}{\text{Mass of Mixture (mg)}} \]
DATA SHEET:  Titrimetric Determination of Sodium Hydrogen Carbonate

Name: ___________________________________________  Sec: ________

Unknown #: Sticker

a. Concentration of NaOH solution from bottle  __________ M.
b. Concentration of HCl solution from bottle  __________ M

c. Wt of vial and initial Mixture  __________ g  __________ g  __________ g  
d. Wt of vial and remaining Mixture  __________ g  __________ g  __________ g  
e. Wt transferred to volumetric flask  __________ g  __________ g  __________ g  
f. Initial HCl buret reading  __________ mL  __________ mL  __________ mL  
g. Final HCl buret reading  __________ mL  __________ mL  __________ mL  
h. Net volume HCl solution  __________ mL  __________ mL  __________ mL  
i. Initial NaOH buret reading  __________ mL  __________ mL  __________ mL  
j. Final NaOH buret reading  __________ mL  __________ mL  __________ mL  
k. Net volume NaOH solution  __________ mL  __________ mL  __________ mL  
l. mmoles HCl initially added  __________ mmol.  __________ mmol.  __________ mmol.  
m. mmoles NaOH titrated  __________ mmol.  __________ mmol.  __________ mmol.  
n. mmoles of HCl remaining  __________ mmol.  __________ mmol.  __________ mmol.  
o. mmole of HCl reacted  __________ mmol.  __________ mmol.  __________ mmol.  
p. mmole of NaHCO₃ in mixture  __________ mmol.  __________ mmol.  __________ mmol.  
q. Mass of NaHCO₃ in mixture  __________ mg  __________ mg  __________ mg  
r. Percent of NaHCO₃ in mixture  __________%  __________%  __________%  

Average percent of NaHCO₃:  __________
Average deviation:  __________
Percent deviation:  __________
1) A student followed the procedure of the exercise and collected the following data for the analysis of a mixture of NaHCO₃ and NaCl.

- Mass of vial + initial mixture = 12.4667 g
- Mass of vial + remaining mixture = 12.3265 g
- Concentration of HCl solution = 0.1020 M
- Concentration of NaOH Solution = 0.1016 M
- Initial HCl buret reading = 0.25 mL
- Final HCl buret reading = 33.50 mL
- Initial NaOH buret reading = 0.48 mL
- Final NaOH buret reading = 21.20 mL

By following the guidelines in the “calculation section” of the exercise, calculate percent of NaHCO₃ in the mixture.

2) If the carbon dioxide is not removed from the solution by boiling, how might the final result be affected? Would the calculated % NaHCO₃ be lower or higher than actual? Explain.

3) Using Equation 1, what volume of 0.1000 M HCl would be required to react completely with 200.0 mg of pure NaHCO₃?