Titrimetric Determination of Sodium Carbonate

Introduction: This experiment involves the titration of sodium carbonate by a strong acid, HCl. This type of analysis is important in determining the buffering capacity of natural waters through a measurement of the carbonate, CO_3^{2-} and bicarbonate, HCO_3^- concentrations. From a knowledge of the pH, the concentrations of H^+, OH^-, H_2CO_3, HCO_3^-, and CO_3^{2-} can be determined in solution (this assumes that there are no other significant weak acids such as H_3BO_3 and H_3PO_4 in the system.) **The equilibria reactions** are:

$H_2CO_3 \rightleftharpoons H^+ + HCO_3^-$	$pK_{a1} = 6.4$
$HCO_3^- \rightleftharpoons H^+ + CO_3^{2-}$	$pK_{a2} = 10.3$
$CO_2(g) + H_2O \rightleftharpoons H_2CO_3$	$pK_g = 1.48$

Before you come to class:

- Record the molar mass of sodium carbonate, Na_2CO_3.
- Write each of the three equilibria reactions (see above).
- Write the analytical reaction (see below) in your lab notebook.
- Record the acid-base indicator that will be used in this experiment as well as the pH transition range and the color change that occurs at that pH (see p. 186 in your book).
- List all of the reagents (formulas and names) in your notebook. Familiarity with the reagents will help you avoid costly mistakes during the experiment.

Reagents: sodium carbonate
0.1000M HCl
bromocresol green indicator

In this experiment a solution of hydrochloric acid will be standardized against pure sodium carbonate and then used to determine the percentage of sodium carbonate in an unknown sample. Reagent grade anhydrous sodium carbonate is suitable for use as a **primary standard** for titrations of strong acids. In this experiment, since the standard is the same compound as the substance being analyzed, determinate errors in the detection of the end-point will be minimized. The **analytical reaction** that will happen during your titration is

$$2\,H^+ + CO_3^{2-} \rightleftharpoons H_2CO_3 \rightleftharpoons H_2O + CO_{2\,(g)}$$

(Cl^- and Na^+ are spectators.)

Note that each mole of carbonate requires two moles of acid for complete titration. Titration to the bromocresol green end-point ensures that all of the carbonate and bicarbonate have been converted to H_2CO_3 and then to H_2O and CO_2.

Summary Procedure: A standard solution of 0.100 M HCl will be provided. You will need about 500 mL of this solution to complete this experiment. The HCl should be standardized against reagent grade (pure) sodium carbonate (a primary standard).

Preparing unknown for pilot titration and drying

1. **Do this before putting your unknown in the oven.** Weigh one 0.45 gram sample of your unknown for titration along with the pure sodium carbonate. **The results of this titration will be used to estimate the sample size of the unknown to be used.** You'll come back to this sample in step #13.
2. Transfer remainder of unknown to weighing bottle. You may label your weighing bottle with your initials and its contents using a Sharpie (this can be removed later using methanol or acetone). Put the bottle and its lid (apart) into a 250 mL beaker and place a small ribbed watch glass on top. Using a Sharpie, label the beaker with your initials and place it in the 110+ °C oven for one hour. Put the weighing bottle and lid into a dessicator immediately after removing from oven, in order to keep it dry as it cools. Once it is cool you may put the lid on the bottle.

Weighing of Primary <u>Standard</u> Na_2CO_3:

3. Note the % Na_2CO_3 (assay) on the reagent bottle. Record this in your notebook. Use this % purity in your calculations.

If the primary standard sodium carbonate was already dried for you, go on to step 6.

4. Dry 1.5 to 2.0 g of pure Na_2CO_3 in a glass weighing bottle in the oven for at least 1 hour. Drying not only removes water but also can reduce bicarbonate impurities.
5. Allow the sodium carbonate to cool in a desiccator.
6. Weigh by difference (to the nearest 0.1 mg) three or four 0.20-0.25 gram portions of the dry material into clean 500 mL Erlenmeyer flasks.

Titration of <u>Standard</u> Sodium Carbonate: perform 3-4 trials.

7. Add about 100 mL of distilled water to each sample of pure sodium carbonate in the 500 mL flask and swirl gently to dissolve the salt.
8. Add enough drops of bromocresol green indicator to one of the flasks so that a strong blue color is observed. Be sure to use the same bottle of indicator each time and add the same number of drops. Titrate with the HCl solution to an intermediate green color.
9. At this point stop the titration and boil the solution gently using a Bunsen burner and tripod stand. Bring the solution to a boil for 2 to 3 minutes, taking care that no solution is lost in the process. Cool to room temperature and wash the inside walls of the flask with distilled water from a wash bottle. Bromocresol green is an example of an indicator whose color strongly depends upon temperature.
10. After allowing the flask to cool, titrate to the yellow endpoint. Titrate to the nearest half-drop. Perform at least three additional replicates. Complete one titration before going on to the next. The boiling step is necessary to yield a sharper endpoint by removing dissolved CO_2 from the solution and forcing the equilibrium

    $HCO_3^- + H^+ \rightleftharpoons H_2CO_3 \rightleftharpoons H_2O + CO_{2\,(g)}$ to be driven to the right.

11. With your titration data, calculate the concentration of the HCl solution. The concentration of your HCl should be very close to 0.1000 M.

"Pilot Titration" of <u>Unknown</u> Sodium Carbonate:

12. Add about 100 mL of distilled water to the <u>pilot</u> unknown sample in the 500 mL flask and swirl gently to dissolve the salt.

13. Add the bromocresol green indicator to the flask and titrate with the HCl solution to the intermediate green color.
14. At this point stop the titration and boil the sample and continue the procedure as was done for the primary standard.
15. Using the number of mL of HCl required in the pilot titration, calculate by ratio the number of grams to be taken for each unknown analysis that would require approximately 35 mL of the 0.100 M HCl.

$$\frac{\text{mL used in pilot titration}}{0.45\ \text{g}} = \frac{35\ \text{mL}}{\text{g to use for titrations}}$$

16. When you calculate the number of grams that would require approximately 35 mL, if you determine that you don't have enough unknown to do three trials, do the following:
    1. Weigh out one sample with the amount you have calculated for 35 mL.
    2. Take the remainder and divide it into two – get the exact weight for each. By doing this, you have 3 samples so you can do 3 trials.

Titration of <u>Unknown</u> Sodium Carbonate: perform 3-4 trials.

17. Add about 100 mL of distilled water to each sample of the unknown sodium carbonate in the 500 mL flask and swirl gently to dissolve the salt.
18. Add the bromocresol green indicator to one of the flasks, and titrate with the HCl solution to an intermediate green color.
19. At this point stop the titration and boil the solution gently using a Bunsen burner and tripod stand. Bring the solution to a boil for 2 to 3 minutes, taking care that no solution is lost in the process. Cool to room temperature and wash the inside walls of the flask with distilled water from a wash bottle.
20. After allowing the flask to cool, titrate to the yellow endpoint. Titrate to the nearest half-drop.

Do your calculations of % Na_2CO_3 as you go. Your result for each trial should be within 1% of each other.

Determination of a <u>Blank</u> (Indicator Correction): perform 3 trials.

21. Determine a blank correction by titrating 100 mL of distilled water to which a spatula tip full of NaCl*(s)* has been added. Be sure to use the same number of drops of bromocresol green indicator used for the previous titrations.
22. Go ahead and heat the sample just like the carbonate titrations. Cool to room temperature
23. Now titrate the blank. Only a few drops of titrant will be needed.
24. Subtract the average blank volume from the <u>total volume delivered in each titration of standard and of analyte.</u>

Report the result in terms of **%Na_2CO_3** in your unknown. The range on unknown values should be between 15% to 55%.