DETERMINATION OF SODA ASH USING HYDROCHLORIC ACID

DISCUSSION: Soda ash is a commercial product containing a high percentage of sodium carbonate (Na_2CO_3). At one time it was obtained by leaching the ashes of sea weeds with water, thus, the name soda ash. At the present time, practically all the sodium carbonate used is made from sodium chloride by the Solvay process. More than 50 percent of the sodium carbonate manufactured in the United States is used in the manufacture of common glass and sodium hydroxide. Other uses include the manufacture of soaps, chemicals, paper, textiles and petroleum products.

Sodium carbonate is neutralized by $HCl_{(aq)}$ in a two-step process described in the following ionic equations:

$$CO_3^{2-}{}_{(aq)} + H^+{}_{(aq)} \rightarrow HCO_3^-{}_{(aq)}$$

$$HCO_3^-{}_{(aq)} + H^+{}_{(aq)} \rightarrow H_2CO_{3(aq)}$$

The two steps in the neutralization are revealed as *two* breaks in the titration curve indicating *two* equivalence points, as shown below in Figure 1.

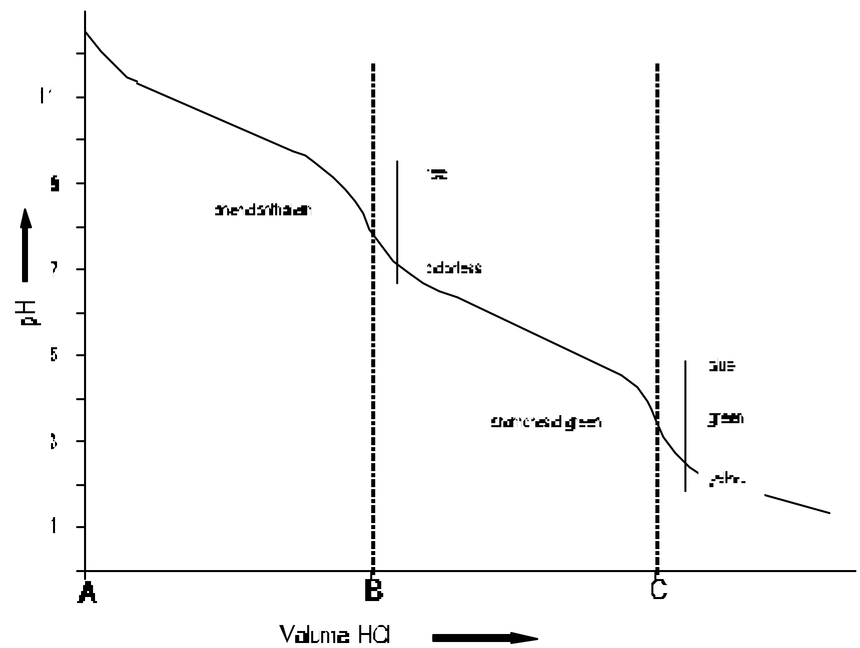

Figure 1

Noting the three letters along the horizontal axis of the titration curve, the carbonate ion in a solution of sodium carbonate exists as $CO_3^{2-}{}_{(aq)}$ at point **A**, principally as $HCO_3^{-}{}_{(aq)}$ at point **B** and predominantly as carbonic acid, $H_2CO_{3(aq)}$, at point **C**. The volumes of $HCl_{(aq)}$ added from point **A** to point **B**, and from point **B** to **C** should be identical, theoretically. Titration with $HCl_{(aq)}$ to the second end point, point **C**, will allow calculation of the total base strength, regardless of whether the base contains NaOH, $NaHCO_3$, Na_2CO_3 or any combination of these. The alkalinity is generally reported in terms of percent Na_2CO_3 in the sample, but it could just as well be reported as percent Na_2O or NaOH. The reported alkalinity in terms of one species or another simply represents the method of computation, and it does not necessarily imply that the base in the sample is in the form reported.

If you know the volume of acid needed to reach the break in the titration curve, point **B**, then you can predict when the second break should occur. Often, the second break, or end point, occurs a bit prematurely because of the carbonic acid which accumulates in solution and lowers the pH. This causes the indicator to change color before the last of the bicarbonate ion is consumed. Fortunately, this problem can be easily overcome by boiling the solution for a few minutes to decompose the carbonic acid and expel the carbon dioxide.

$$H_2CO_{3(aq)} \rightarrow H_2O_{(l)} + CO_{2(g)}$$

As the solution is boiled and the carbonic acid is destroyed, the pH rises and the remaining bicarbonate ion will cause the solution to be mildly alkaline. This is clearly shown as the color of the indicator changes back as the solution is heated. After the heating is over, the solution is cooled and the titration completed. This time however, the pH change accompanying the final additions of acid is considerably larger and, as a consequence, allowing the end point to be located more accurately.

PREPARATION OF REAGENTS:

0.1 N HCl(aq) Prepare 2 liters of approximately 0.1 N $HCl_{(aq)}$ from concentrated hydrochloric acid which has a density of 1.19 g/mL and is between 37 and 38 % $HCl_{(aq)}$ by weight. *Shake the prepared solution well* now and before each use. The standardized acid may be used in a later experiment, so do not dispose of it when finished with this analysis unless told to do so by your instructor.

EXPERIMENTAL PROCEDURE:

Standardization of $HCl_{(aq)}$

Dry a sample of primary-standard Na_2CO_3 for 1 ! 2 hours at 110°C and cool in a desiccator. Weigh out 4 samples of 0.2000 g, weighed accurately to four places after the decimal. Dissolve each sample in about 50 mL of deionized water (boiled water is not necessary). Add 3 drops of phenolphthalein and titrate until the solution just loses its pink color. *Note this approximate volume as that of the* ***first*** *end point.* Add 3 drops of bromcresol green and, without refilling the buret, continue to titrate until the blue color of the indicator just disappears. Once the blue color is gone, the resulting color may be the intermediate green (which is preferred) or yellow. Boil the solution for about 3 minutes, cool to room temperature under tap water and complete the titration. During the heating process the indicator should change back to blue. If it does not, the solution was over-titrated (too much acid was added) and it should be discarded. Record the final volume and calculate the normality of $HCl_{(aq)}$ to four significant figures. The expected tolerance is ±0.0005.

Titration of Na_2CO_3 in Unknown Sample

Dry an unknown soda ash sample for 1-2 hours at 110°C and cool in a desiccator. Weigh out 4 samples of about 0.5 g each, weighed accurately to four decimal places. Dissolve each in about 50 mL of unboiled deionized water and proceed as above. Calculate the percent of Na_2CO_3 in each sample. The range of percentages for the four trials should not exceed 0.3%.

(NOTE: Save the acid for the mixed acid experiment unless directed otherwise.)

REPORT SHEET: **DETERMINATION OF SODA ASH USING HYDROCHLORIC ACID**

Name ____ Date: ____________ Sample No. ____________

Please print; last name first

Standardization of $HCl_{(aq)}$

	Trial 1	Trial 2	Trial 3	Trial 4
mass of standard Na_2CO_3				
volume $HCl_{(aq)}$				
$N_{HCl(aq)}$				

Average normality of $HCl_{(aq)}$ (4 sig. figs.): ____________________

Circle all values used to determine the average normality of $HCl_{(aq)}$.

Analysis of the Unknown Na_2CO_3 Sample

	Sample 1	Sample 2	Sample 3	Sample 4
mass of unknown Na_2CO_3 sample				
volume $HCl_{(aq)}$				
% Na_2CO_3 in sample				

Average % Na_2CO_3 in sample (4 sig. figs.): ____________________

Circle all values used to obtain the average % Na_2CO_3 reported above.

Show the calculation clearly for one trial and one sample on the back of this page.