Ima Student
January 28, 2022
CHE 131 – Section 201

Experiment 1 – Determination of the Alkalinity of Soda Ash

Abstract:

Titrimetric analysis is used to determine the concentration of a species in an unknown solution by reacting it with another solution of known concentration. Here, a standardized hydrochloric acid solution was used to calculate the mass percent of sodium carbonate present in an unknown sample of soda ash. The hydrochloric acid titrant was first standardized using 0.1344 M sodium hydroxide and a phenolphthalein indicator. Aliquots of a stock aqueous solution of soda ash containing an unknown amount of sodium carbonate were titrated against the standardized 0.99 M hydrochloric acid until a bromocresol green endpoint was reached. Through the volume of acid dispensed, the molarity of the titrant, and the stoichiometric ratio of sodium carbonate to hydrochloric acid, the moles of sodium carbonate present in the aliquot were calculated. This was then converted to a mass using the molar mass of sodium carbonate. Proportional reasoning and the mass of soda ash in the stock solution were used to find the overall mass percent of sodium carbonate present in unknown #248, (27.1 ± 0.2)%, which is larger than the literature value of 26.28%.

Procedure:

The procedure can be found in: CHE 131 Experiment 1, General Chemistry I Lab, Autumn Quarter 2020-2021, DePaul University. [**Online**] https://www.d2l.depaul.edu (accessed on September 9, 2020).

- There were no deviations from the procedure cited above.

Data and Results:

Crude sodium carbonate, known as soda ash, contains an unknown amount of sodium carbonate in addition to traces of other related compounds. Because an aqueous solution of sodium carbonate is basic, titrimetric analysis lends itself as a viable technique to calculate the mass percent of sodium carbonate present in a sample of soda ash. This requires that the concentration of the titrant, which is hydrochloric acid in this case, is accurately known. Concentrated hydrochloric acid was used to create a dilute solution of hydrochloric acid, which was standardized with a 0.1344 M aqueous sodium hydroxide solution. Three 25.00 mL aliquots of this sodium hydroxide solution were titrated against the hydrochloric acid solution until the characteristic pink color of phenolphthalein disappeared in each case (Table 1).

Table 1: Standardization of HCl Titrant Using 25.00 mL Aliquots of 0.1344 M NaOH

Trial	**Initial Burette Reading (mL)**	**Final Burette Reading (mL)**	**Volume HCl Added (mL)**	**Concentration HCl (M)**
1	9.40	44.98	35.58	0.09444
2	6.85	40.00	33.15	0.1014
3	6.02	39.33	33.31	0.1009

The volume V_{HCl} of hydrochloric acid necessary to reach the endpoint in Trial 1 was given by the difference between the final and initial burette readings, V_{f} and V_{i}, respectively (Equation 1).

$$V_{\mathrm{HCl}} = V_{\mathrm{f}} - V_{\mathrm{i}} \tag{1}$$

Because sodium hydroxide and hydrochloric acid react in a 1:1 molar ratio at the equivalence point, as seen in the balanced chemical equation below,

$$\mathrm{HCl}_{(aq)} + \mathrm{NaOH}_{(aq)} \rightarrow \mathrm{H_2O}_{(\ell)} + \mathrm{NaCl}_{(aq)}$$

the moles of hydrochloric acid added is equal to the moles of sodium hydroxide originally present. This value is equal to the product of the volume V_{NaOH} and concentration M_{NaOH} of the sodium hydroxide aliquot. The molarity M_{HCl} of hydrochloric acid in Trial 1 was calculated from the moles of acid added and the volume of acid necessary to reach the endpoint (Equation 2).

$$M_{\mathrm{HCl}} = \frac{V_{\mathrm{NaOH}} M_{\mathrm{NaOH}} \left(\dfrac{1 \text{ mol HCl}}{1 \text{ mol NaOH}} \right)}{V_{\mathrm{HCl}}} \tag{2}$$

The results from the three trials were averaged together to give the concentration of the hydrochloric acid titrant, (0.099 ± 0.004) M.

After standardizing the acid solution, a sample of soda ash was analyzed to determine the amount of sodium carbonate present. A stock solution was made by dissolving 5.0249 g of soda ash from vial #248 in enough deionized water to make 250.0 mL of solution. The soda ash, which is hygroscopic, was placed in a dessicator overnight to ensure any water present in the sample was removed. A 25.00 mL aliquot of this stock solution was titrated against the standardized HCl solution with bromocresol green used as an indicator. The pH of the soda ash analyte was tracked throughout the titration using a calibrated pH meter (Figure 1).

The titration curve for Trial 1 shows two sharp decreases in pH: one centered at 13.10 mL and the other centered at 26.20 mL. This is due to the 2:1 stoichiometric ratio of hydrochloric acid and sodium carbonate in the reaction of interest:

$$2\mathrm{HCl}_{(aq)} + \mathrm{Na_2CO}_{3\,(aq)} \rightarrow \mathrm{H_2O}_{(\ell)} + \mathrm{CO}_{2\,(g)} + 2\mathrm{NaCl}_{(aq)}$$

When hydrochloric acid was first added to the sodium carbonate analyte, hydrogen carbonate was formed from the reaction between hydronium and carbonate. Hydrogen carbonate continued to form until all of the carbonate had reacted, which occurred at the first equivalence point of the titration when a total of 13.10 mL of hydrochloric acid was added. The newly formed hydrogen carbonate was converted to carbonic acid, which is unstable in solution and decomposes to form water and carbon dioxide gas, as additional hydrochloric acid was introduced to the analyte. Therefore, all of the carbonate initially present in the analyte was fully converted to water and carbon dioxide at the second equivalence point, when a total volume of 26.20 mL of 0.099 M hydrochloric acid was added.

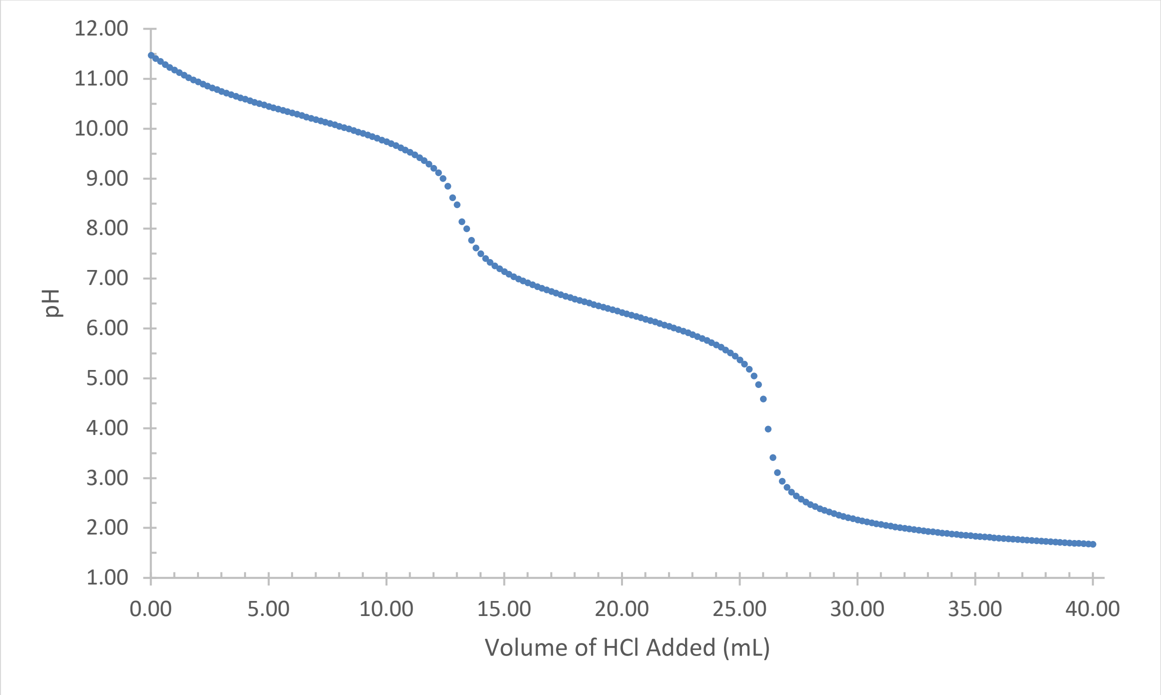

Figure 1. Representative titration curve for the reaction between 0.099 M HCl and a 25.00 mL aliquot of soda ash sample #248. All of the carbonate present is fully reacted to form water and carbon dioxide at 26.20 mL, which corresponds to the midpoint of the second sharp decrease in pH.

At the second equivalence point, a stoichiometric amount of hydrochloric acid had been added to the sodium carbonate analyte. The number of moles of acid added was equal to the product of the volume V_{HCl} of hydrochloric acid necessary to reach the second equivalence point and the molarity M_{HCl} of the titrant. The mass $m_{\text{Na}_2\text{CO}_3}$ of sodium carbonate present in the aliquot for Trial 1 was calculated using this value along with the stoichiometric ratio between hydrochloric acid and sodium carbonate and the molar mass $MM_{\text{Na}_2\text{CO}_3}$ of sodium carbonate (Equation 3).

$$m_{\text{Na}_2\text{CO}_3} = V_{\text{HCl}} M_{\text{HCl}} \left(\frac{1 \text{ mol Na}_2\text{CO}_3}{2 \text{ mol HCl}} \right) MM_{\text{Na}_2\text{CO}_3} \tag{3}$$

The stock soda ash solution was mixed well to ensure it was uniform. Therefore, the concentration of soda ash in each aliquot was equal to the concentration of the stock solution. The mass m_{aliquot} of soda ash in each aliquot was calculated from this proportionality using the volume V_{aliquot} of each aliquot, the mass m_{stock} of soda ash in the stock, and the volume V_{stock} of the stock solution (Equation 4).

$$m_{\text{aliquot}} = \left(\frac{m_{\text{stock}}}{V_{\text{stock}}} \right) V_{\text{aliquot}} \tag{4}$$

Because the stock solution was uniform in concentration throughout, the mass percent $\%_{\text{Na}_2\text{CO}_3}$

each aliquot, which is related to the quotient of the mass $m_{\mathrm{Na_2CO_3}}$ of sodium carbonate and the mass m_{aliquot} of soda ash present in the aliquot (Equation 5).

$$\%_{\mathrm{Na_2CO_3}} = \left(\frac{m_{\mathrm{Na_2CO_3}}}{m_{\mathrm{aliquot}}}\right) \times 100\% \quad (5)$$

The results for each trial (Table 2) were calculated independently and then averaged together to give the mass percent of sodium carbonate in sample #248, (27.1 ± 0.2)%.

Table 2: Titrimetric Analysis of Sodium Carbonate in Soda Ash Sample #248

Trial	**Volume HCl Added (mL)**	**Mass Na_2CO_3 (g)**	**% Na_2CO_3**
1	26.20	0.1373	27.32
2	25.81	0.1353	26.92
3	26.01	0.1363	27.13

Discussion:

The experimental result of (27.1 ± 0.2)% generally agrees with the expected mass percent of sodium carbonate in sample #248, which is 26.28%. Although the standard deviation for the three trials was relatively small, implying a high degree of precision, the experimental mass percent was larger than the literature value in every trial. During the experiment, Trial 1 was possibly overtitrated because the yellow endpoint due to the bromocresol green indicator was difficult to discern. If a larger volume of titrant were added than necessary, this would imply a larger amount of carbonate present in the analyte because more acid was necessary to neutralize all of the carbonate present. Assuming all of the carbonate in the sample was due to sodium carbonate, this would directly lead to a larger mass percent of sodium carbonate in the soda ash sample.

An artificially inflated value for the concentration of the sodium hydroxide solution used in the standardization of the hydrochloric acid titrant would also lead to consistently larger than expected amounts of carbonate in each trial. Solid sodium hydroxide is hygroscopic, meaning it takes on moisture from the air. Therefore, when making the sodium hydroxide standard, the actual mass of sodium hydroxide in the solution was less than what was recorded, which corresponded to the mass of both sodium hydroxide and water. Both this and the increase in the total volume of the solution due to the water present in the solid would lead to a hydroxide concentration that was actually smaller than the calculated value. If this were the case, it would imply the hydrochloric acid was more concentrated that it truly was because the endpoint was reached earlier than expected. If the true concentration of the standardized hydrochloric acid were less than 0.099 M, a larger volume of titrant than expected would be necessary to fully react with all of the carbonate present in the individual aliquots of soda ash solution. Similar to the effect of overtitrating, a larger volume of titrant implies a larger mass percent of sodium carbonate in the sample.

A known potential complication in this experiment is the production of carbonic acid as hydrochloric acid was added to the soda ash. If the carbon dioxide gas produced by the decomposition of carbonic acid were not allowed to leave the analyte vessel, the pH of the system would be lower than expected, thus causing the indicator to change color earlier than expected.

Correspondingly, a smaller volume of titrant necessary to reach the endpoint would be recorded. Because the volume of titrant is proportional to the mass of sodium carbonate present, the calculated mass percent of sodium carbonate in the unknown would be smaller than the literature value. This complication can be avoided by boiling the soda ash analyte to drive off any lingering dissolved carbon dioxide after the endpoint was first achieved. Although the sample was not boiled during this experiment, it did not appear to be a confounding effect as the calculated mass percent of sodium carbonate in each trial was larger than the literature value, not smaller.