<u>**Experiment 2**</u>

Determination of the Amounts of Sodium Hydroxide Solution and Sodium Carbonate in a Mixed Solution

<u>**Date of Experiment:**</u> 21/9/2005
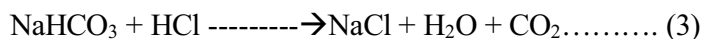
<u>**Objective:**</u> To determine the percentage by mass of Sodium Hydroxide and Sodium Carbonate in a mixed solution.

<u>**Introduction**</u>

Sodium carbonate and sodium hydroxide are both alkaline in nature. When hydrochloric acid is added to the mixture, sodium hydroxide reacts like this:

$$NaOH + HCl \longrightarrow NaCl + H_2O \dots\dots\dots\dots\dots\dots\dots (1)$$
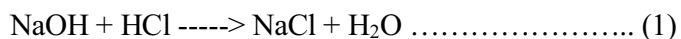
Sodium carbonate reacts with hydrochloric acid in a similar way but the reaction comes in 2 steps:

$$Na_2CO_3 + HCl \dashrightarrow NaHCO_3 + NaCl\dots\dots\dots\dots (2)$$

$$NaHCO_3 + HCl \dashrightarrow NaCl + H_2O + CO_2\dots\dots\dots (3)$$

The pH conditions in which reactions (1) and (2) take place are approximately the same: they both occur at a relatively higher pH (at a pH value higher than 7). Reaction (3) is quite different from the other 2 reactions. It takes place at a lower pH (at a pH value lower than 7).

So, using the 2 acid-base indicators, the end points for the reactions can be found. Phenolphthalein turns from pink to colorless as the pH value changes from 10 to 8. Methyl orange turns from yellow to orange when the pH value drops from about 4 to 3.

After adding phenolphthalein and then titrating the solution, the end point for reactions (1) and (2) can be found. If we further add methyl orange and titrate the mixture afterwards, the end point for reaction (3) is also found. By considering the amount of hydrochloric acid used for both titrations, the percentage by mass of sodium carbonate and sodium hydroxide can be found.

This is just one of the methods to determine the percentage by mass of sodium carbonate and sodium hydroxide. We can make use of barium chloride in this case. This is because when barium ions and carbonate ions come in contact, the following reaction occurs.

$$Ba^{2+} (aq) + CO_3^{2-} (aq) \text{-----------} \rightarrow BaCO_3 (s)$$
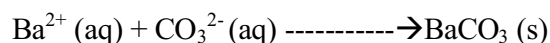
How can we utilize this special point of barium ions? We can use this to precipitate out the carbonate ions. Then what would be left in the mixture would be sodium hydroxide only. Then, using phenolphthalein as an indicator, we could find out the number of moles of sodium hydroxide in the solution.

Then one may ask,' How do I find out the percentage by mass of sodium carbonate using the second method?' We can do so by titrating the pure solution (no barium chloride added) using methyl orange indicator. The end point indicates the amount of hydrochloric acid used to neutralize both sodium carbonate and sodium hydroxide. The total number of moles of both sodium carbonate and sodium hydroxide can be found. By this way, we can find the percentage by mass of the both substances.

Procedure

1. All the apparatus involved were washed beforehand.

Method 1

2. With a pipette, 25cm^3 of the solution mixture was transferred into a conical flask.
3. Appropriate amount of hydrochloric acid of was poured into a burette, and the initial reading was taken.
4. Using phenolphthalein as an indicator, the solution in the conical flask was titrated by the hydrochloric acid. The titration stopped when the color of the indicator changes from pink to colorless. The reading was taken.
5. Methyl orange was further added to the solution. Titrate the solution until the color changed from yellow to orange. The readings were taken.
6. The conical flask was washed and the burette was filled with hydrochloric acid.
7. Steps 2 to 6 were repeated for 4 more times.

Method 2

8. 25 cm^3 of the solution mixture was transferred to the conical flask with a pipette.
9. Hydrochloric acid was added into the burette, with the initial readings taken.
10. Using methyl orange as indicator, the solution in the conical flask was titrated until the color changes from yellow to orange. The readings were taken.
11. The conical flask was washed with tap water and rinsed by distilled water.
12. Steps 8 to 11 were repeated for 2 more times.
13. 25 cm^3 of the solution mixture was transferred to the conical flask with a pipette.
14. Barium chloride solution was poured into the conical flask until no more white solids were formed.
15. Hydrochloric acid was filled into the burette. The initial reading was taken.
16. Using phenolphthalein as indicator, the solution in the conical flask was titrated until the color changed from pink to colorless.

17. The final readings were taken.

18. Steps 13 to 17 were repeated for 2 more times.

Data and Calculation

Method 1

	1	2	3	4	5
Initial reading	3.3 cm^3	2.5 cm^3	0.1 cm^3	1.5 cm^3	3.5 cm^3
Reading when color changes from pink to colorless (Volume of HCl used)	17.2 cm^3 (13.9 cm^3)	17.2 cm^3 (14.7 cm^3)	13.7 cm^3 (13.6 cm^3)	15.4 cm^3 (13.9 cm^3)	17.5 cm^3 (14 cm^3)
Reading when color changes from yellow to orange (Volume of HCl used)	22.5 cm^3 (5.3 cm^3)	21.5 cm^3 (4.3 cm^3)	18.7 cm^3 (5.0 cm^3)	20.5 cm^3 (5.1 cm^3)	22.7 cm^3 (5.2 cm^3)

(The 1st and 2nd titration were rejected as the 1st was a trail and the 2nd was inconsistent with other results)

Average volume of HCl used to neutralize both NaOH and Na$_2$CO$_3$ into water and hydrogencarbonate ions: $(13.6 + 13.9 + 14) /3 = 13.833$ cm^3

Number of moles of HCl in this step: $13.833/1000 \times 0.09868 = 1.365 1 \times 10^{-3}$ mol

So, the number of OH$^-$ and CO$_3^{2-}$ adds up to 1.3651×10^{-3} mol.

Average volume of HCl used to neutralize hydrogencarbonate ions = $(5.0 + 5.1 + 5.2) /3 = 5.1$ cm^3

Number of moles of HCl in this step: $5.1/1000 \times 0.09868 = 5.033 \times 10^{-4}$ mol.

So, there are also 5.033×10^{-4} moles of Na$_2$CO$_3$ in the experiment.

Mass of Na$_2$CO$_3$ in the experiment:

$$5.033 \times 10^{-4} \times (23 \times 2 + 12 + 16 \times 3) = 0.5335g$$

Mass of NaOH in the experiment:

$$(1.3651 \times 10^{-3} - 5.033 \times 10^{-4}) \times (23 + 17) = 0.03442g$$

So, the percentages by mass of NaOH and Na$_2$CO$_3$ are 39.15% and 60.85% respectively.

Method 2

A table showing the volume of HCl used for reacting both Na_2CO_3 and NaOH

	1	2	3
Final reading	22.6 cm³	40.8 cm³	19.7 cm³
Initial reading	3.1 cm³	21.6 cm³	0.5 cm³
Volume of HCl used	19.5 cm³	19.2 cm³	19.2 cm³

(Due to time limitations, only 3 titrations could be done and the first titration is not rejected)

Average volumes of HCl used = (19.5 + 19.2 X 2)/ 3 = 19.3 cm³

No of moles of HCl used = 19.3/1000 X 0.09868 = 1.905×10^{-3} moles

A table showing the volume of HCl used for reacting NaOH.

	1	2	3
Final reading	27.7 cm³	35.5 cm³	43.6 cm³
Initial reading	19.7 cm³	27.7 cm³	35.5 cm³
Volume of HCl used	8 cm³	7.8 cm³	8.1 cm³

(Due to time limitations, only 3 titrations could be done and the first titration is not rejected)

Average volumes of HCl used = (8 + 7.8 + 8.1)/3 = 7.967 cm³

No of moles of NaOH in the titration = 7.967/1000 X 0.09868 = 7.862×10^{-4} mol

No. of moles of HCL used to titrate Na_2CO_3 =
$$1.905 \times 10^{-3} - 7.862 \times 10^{-4} = 1.12 \times 10^{-3} \text{ mol}$$

So, the no. of mole of $Na_2CO_3 = 1.12 \times 10^{-3}/2 = 5.6 \times 10^{-4}$ mol.

Mass of NaOH = 7.862×10^{-4} X (23 + 17) = 0.03145g

Mass of $Na_2CO_3 = 5.6 \times 10^{-4}$ X (46 + 12 + 16X3) = 0.0593 g

So, the percentages by mass of NaOH and Na_2CO_3 are 34.7% and 65.3% respectively.

Conclusion

In method 1, the percentages by mass of NaOH and Na_2CO_3 are 39.15% and 60.85% respectively.

In method 2, the percentages by mass of NaOH and Na_2CO_3 are 34.7% and 65.3% respectively.

Discussion

The results obtained from both experiments were not quite consistent. The reasons are to be discussed below.

Firstly, many human errors have occurred in the titration. For example, while shaking the solution inside the conical flask, I accidentally spilt a drop onto the table. A smaller amount of HCl would be required to reach the end point in this case. This is because a smaller amount of substance is actually being titrated.

Also, unlike the first titration, this experiment involves more end points to be determined. In the first TAS, the end point only needs to be determined once every titration. But this time, two determinations need to be made for every titration in the first method. If there are no definitions of 'orange' or 'colorless', which is actually the case, inconsistency and thus error will occur.

Furthermore, this is my first time using phenolphthalein to do titrations since the start of F.6. I forgot the fact that after the end point is reached, which is colorless, further adding HCl into the conical flask will not change the color of the solution. The solution will remain colorless. Errors will occur in this case. I may take the final readings , which an excess of HCl is added, and the readings would be wrong. The readings would be higher than the actual amount of HCl required to reach the end point.

So, due to above reasons, many errors were made in this experiment. Then how may I improve my experiment?

I figured out that the main reason for me to make so many errors was due to my poor time management. While others were already starting method 2, I was still working hard on method 1. I have used an unreasonably long time to wash the apparatus and my motion was too slow. If I could allocate my time appropriately, I would not have made so many errors.

Another way to improve is to use a pH meter instead of using an indicator to determine the equivalence point. The equivalence point would be determined more accurately using pH meter.

How do I know when to stop adding $BaCl_2$ in method 2? My method is simple: add $BaCl_2$ until no more white solid forms. I tilted the conical flask, and then I added $BaCl_2$ using a dropper, dropping the Barium solution onto the side of the flask. If no white solid is formed, then I had finished adding $BaCl_2$.

Answer To Study Questions

1. **Explain the changes of colors of the indicators in the above titrations.**

   In method 1, the solution in the conical flask consists of NaOH and Na_2CO_3, which are basic. So, when phenolphthalein is added to the solution, the solution turns pink. The pH of the solution drops when HCl is added to the conical flask and the end point is reached. So, phenolphthalein eventually turns colorless.

   In the second step of this method, methyl orange is added. As the solution in the

conical flask is still alkaline, methyl orange is yellow at the beginning. When HCl is added to the conical flask and the end point is reached, the pH drops and the indicator turns from yellow to orange.

In method 2, there are two titrations. In the one without $BaCl_2$ added, methyl orange was added initially. As the solution in the conical flask was alkaline, methyl orange turned yellow at the start. When HCl was added and the end point was reached, the solution become acidic and the indicator turned orange in color.

In the titration with $BaCl_2$ added into the conical flask, phenolphthalein was added. Initially, the solution in the flask was alkaline. So, the indicator was pink at the start. After the end point was reached by adding HCl into the flask, the solution had a lower pH and the indicator turned to colorless.

2. Explain why the titration should be carried out slowly and with constant stirring in (b).

This can make sure that all sodium hydroxide have reacted before further adding hydrochloric acid into the conical flask. After adding barium chloride was added into the conical flask, precipitates were formed. These precipitates would hinder the reaction of sodium hydroxide and hydrochloric acid. So, in order to allow all the hydrochloric acid to be reacted before anymore of it is added, we need to stir it and do it slowly.

3. Suggest with reasons which method is more accurate in the experiment.

I think that method 2 is more accurate. This is because method 1 involves 2 end points in 1 titration. If an error was made in the first part of the titration in method 1, the results obtained in the second part would also be wrong.

Also, CO_3^{2-} reacts in 2 steps. But how will I be able to confirm that some HCO_3^{2-} has not further reacted to form carbon dioxide and water in the first part? This also lowers the accuracy of method 1.