CHEMISTRY

10^{th} and 11^{th} class Date : 27-09-2021

Dr. K. Ananthanarayanan
Associate Professor (Research)
Department of Chemistry
Room No 319, 3^{rd} Floor, Raman Research Park

Email : ananthak@srmist.edu.in
Phone : 9840154665

Determination of Na_2CO_3 and NaOH in a mixture by titration

Expt. No. : 1

Experiment

- **Aim :**

To determine the amount of Na_2CO_3 and NaOH in a mixture using hydrochloric acid.

- **Apparatus required:**

Conical flask,100 mL standard flask, 20 mL pipette burette, funnel, glass rod.

- **Chemicals required:**

Hydrochloric acid, phenolphthalein indicator, methyl orange indicator, distilled water and mixture solution.

Water standard, IS 10500:2012

Table 1 Organoleptic and Physical Parameters
(*Foreword* and *Clause* 4)

Sl No.	Characteristic	Requirement (Acceptable Limit)	Permissible Limit in the Absence of Alternate Source	Method of Test, Ref to Part of IS 3025	Remarks
(1)	(2)	(3)	(4)	(5)	(6)
i)	Colour, Hazen units, *Max*	5	15	Part 4	Extended to 15 only, if toxic substances are not suspected in absence of alternate sources
ii)	Odour	Agreeable	Agreeable	Part 5	a) Test cold and when heated b) Test at several dilutions
iii)	*p*H value	6.5-8.5	No relaxation	Part 11	—
iv)	Taste	Agreeable	Agreeable	Parts 7 and 8	Test to be conducted only after safety has been established
v)	Turbidity, NTU, *Max*	1	5	Part 10	—
vi)	Total dissolved solids, mg/l, *Max*	500	2 000	Part 16	—

NOTE — It is recommended that the acceptable limit is to be implemented. Values in excess of those mentioned under 'acceptable' render the water not suitable, but still may be tolerated in the absence of an alternative source but up to the limits indicated under 'permissible limit in the absence of alternate source' in col 4, above which the sources will have to be rejected.

Acid-base titration

- A **quantitative analysis** of acids and bases; through this process, an acid or base of **known concentration** neutralizes an acid or base of **unknown concentration.**
- The titration progress can be monitored by **visual indicators**
- The reaction's **equivalence point** is the point at which the titrant has **exactly neutralized** the acid or base in the unknown analyte; if you know the volume and concentration of the titrant at the equivalence point, **you can calculate the concentration of a base or acid in the unknown solution**.

5

Acid-base indicators

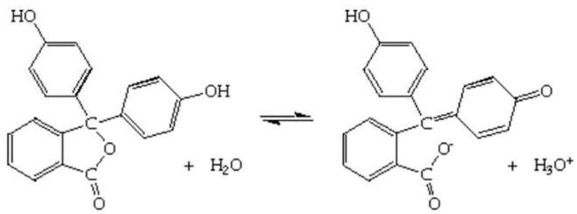
- Acid - base indicators (also known as pH indicators) are substances which change color with pH.
- They are usually weak acids or bases
- Consider an indicator which is a weak acid, with the formula HI. At equilibrium, the following chemical equation is established

$$HI + H_2O \rightleftharpoons H_3O^+ + I^-$$

- The acid, HI, and the conjugate base, I^-, have different colors.
- Common examples include phenolphthalein and methyl orange.

9/29/2021 6

Phenolphthalein indicator



colourless (Acid) pink (Base)

- Under acidic conditions, the equilibrium is to the left, and the concentration of the anions is too low for the pink colour to be observed. (**phthalein class of dye**)
- However, under alkaline conditions, the equilibrium is to the right, and the concentration of the anion becomes sufficient for the pink colour to be observed. **pH range : 8 – 9.8**

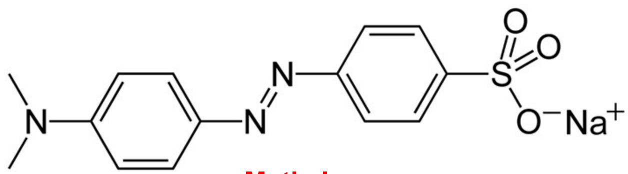
Phenolphthalein indicator contd..

- As an indicator of a solution's pH, phenolphthalein is **colorless below pH 8.2** and attains a **pink to deep red hue above pH 9.0.**

Increasing pH

Methyl orange indicator

- Methyl orange ($C_{14}H_{14}N_3NaO_3S$) is a water soluble **azo dye** that shows color change in the **pH range of 3.1 to 4.4**
- Because it changes color at the pK_a of a mid strength acid, it is usually used in titration for acids
- Methyl orange has a pK_a of 3.47 in water at 25 °C



Methyl orange

9/29/2021 9

Methyl orange indicator contd..

- Methyl orange is a pH indicator frequently used in titration because of its clear and distinct color variance at different pH values. Methyl orange shows red color in acidic medium and yellow color in basic medium

Decreasing pH

9/29/2021 10

Principle (double indicator)

- The titration of a mixture of NaOH and Na_2CO_3 with a standardized HCl solution has two **equivalence points.**
- The first equivalence point is due to the conversion of all the carbonate to bicarbonate and sodium hydroxide to sodium chloride as follows:

$$NaOH + HCl \rightarrow NaCl + H_2O$$

$$Na_2CO_3 + HCl \rightarrow NaHCO_3 + NaCl$$

- The pH of the resulting solution is around 8 therefore phenolphthalein could be used as an indicator.
- At the end point, all the hydroxide ions (OH^-) and only half of the carbonate ions (CO_3^{2-}) are reacted with hydrochloric acid.

AN1

- As can be seen, when we go on adding more and more of HCl, the pH of the solution keeps on falling.
- When Na_2CO_3 is converted to $NaHCO_3$, completely, the solution is weakly basic due to the presence of $NaHCO_3$ (which is a weaker base as compared to Na_2CO_3).
- At this instant phenolphthalein changes colour since it requires this weakly basic solution to change its colour.
- Therefore, remember that phenolphthalein changes colour only when the weakly basic $NaHCO_3$ is present.

Slide 12

AN1 Ananth Narayanan, 21-04-2021

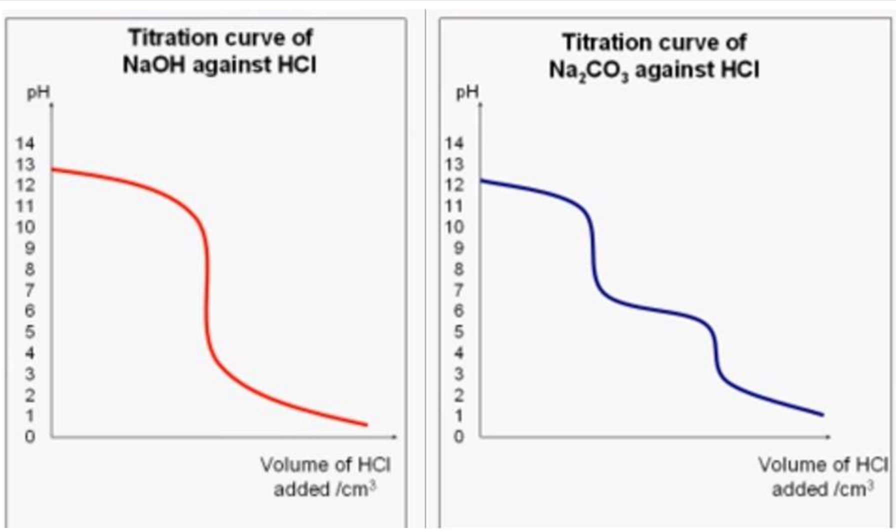
Principle contd..

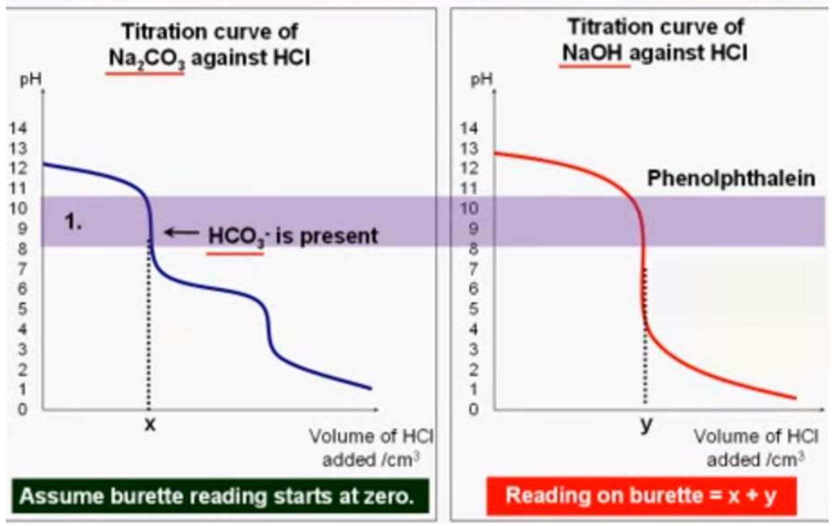
- The second equivalence point is due to the reaction of the resulting bicarbonate with an excess of HCl solution as follows:

$$NaHCO_3 + HCl \rightarrow NaCl + CO_2 + H_2O$$

- In this stage methyl orange is used to find the equivalence point because the pH of the solution at this point will be around 3.8

- When the titration is continued with methyl orange indicator, the remaining half of CO_3^{2-} ions will be neutralized with HCl at the end point





Procedure

Titration I : Standardization of HCl

- Pipette out **20 ml of 0.05 N Na_2CO_3** solution into a clean conical flask and add 2-3 drops of methyl orange indicator to the solution.
- Then titrate the solution against hydrochloric acid taken in the burette.
- Record end point (burette reading) when color changes from yellow to orange (orange red).
- Repeat the titration till the concordant (two consecutive burette readings exactly same) value is obtained.

***Sodium hydroxide* NaOH** is not used as a *primary standard* solution *Why*?

$$2NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O$$

So, the commercial sample of solid sodium hydroxide contains a quantity of Na_2CO_3

Commercially available NaOH contains impurities of Na_2CO_3, and also readily absorbs H_2O from the atmosphere

Procedure

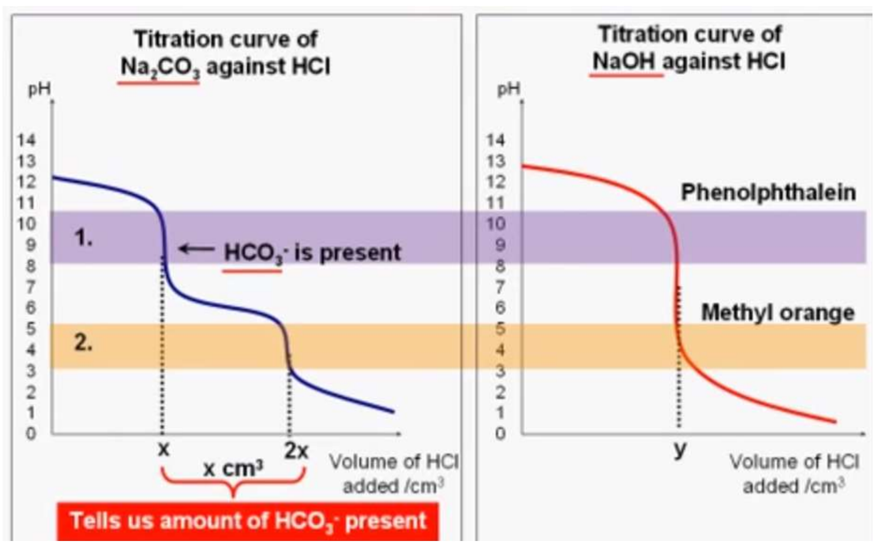
Titration II: Estimation of Na_2CO_3 and NaOH in a given mixture

- Dilute the given unknown solution to **100 ml** in a standard flask using distilled water.
- Pipette out **20 ml** of this made up solution into a clean conical flask. Add 2-3 drops of phenolphthalein indicator to the solution and titrate against standardized HCl.
- Record the burette reading as an end point (phenolphthalien end point) of the titration when **disappearance of pink color is observed.**
- Consider the burette reading at the end point be **'A'** ml.

Procedure

Titration II: Estimation of Na_2CO_3 and NaOH in a given mixture

- To the same solution, add 2-3 drops of methyl orange indicator and continue the titration till the color changes from **yellow to orange**
- Note down the burette reading as methyl orange end point. Consider it to be **'X'** ml.
- Repeat the titration till the concordant (two consecutive burette readings exactly same) **value for A and X is obtained**.



Calculation (principle)

- **A** = all hydroxide ions + half of carbonate ions (Volume of HCl used for NaOH neutralization + Volume of HCl used for half Na_2CO_3 neutralization)
- **B** = half the carbonate ions after phenolphthalein end point (Volume of HCl for half neutralization of Na_2CO_3) **B=X-A**
- **2B** = all carbonate ions (Volume of HCl used for total neutralization of Na_2CO_3)
- **A-B** = all hydroxide ions (Volume of HCl used for total neutralization of NaOH)

Tabular column

Titration 1 : Standardization of HCl

S.No.	Volume of sodium carbonate solnution (ml)	Burette reading		Concordant Value	Indicator
		Initial	Final		
					Methyl orange (2-3 drops)

Calculation

Volume of HCl = V_1 ml (end point)

Normality of HCl = ----- ? N_1

Volume of Na_2CO_3 (V_2) = 20 ml

Normality of Na_2CO_3 (N_2) = 0.1N **0.05 N**

Normality of HCl (N_1) = $(20 \times 0.1) / V_1$

= ______ N

Tabular column

Titration II: Estimation of Na_2CO_3 and NaOH in a given mixture.

Sl. No.	Volume of pipette solution (mL)	Burette Reading (mL)			Concordant Value	
		Initial	Vol. consu. Phenolphthalein end point (A)	Vol. consu. Methyl orange end point (X)	Phenolphthalein	Methyl orange

Vol. consumed for methyl orange end point after phenolphthalein end point B = X-A

Calculation

I Estimation of the amount of Na_2CO_3

Volume of HCl V_1 = 2B (B is the titre value, **after** phenolpthalein end point)

Normality of HCl = N_1 (from Titration I)

Volume of mixture V_2 = 20 ml

Normality of Na_2CO_3 N_2 = $V_1 N_1 / V_2$

= $2B \times N_1 / 20$

Amount of Na_2CO_3 present in whole of the given solution = $\frac{N_2 \times 53 \text{ (Eq. wt. of } Na_2CO_3)}{10}$

9/29/2021 27

Calculation

II Estimation of amount of NaOH:

Volume of HCl V_1 = (A-B) ml

Normality of HCl (N_1) = N_1 (from Titration I)

Volume of mixture V_2 = 20 ml

Normality of NaOH $\mathbf{N_2}$ = $V_1 \times N_1 / 20$

= $(A-B) \times N_1 / 20$

Amount of NaOH present in whole of the given solution = $\frac{N_2 \times 40 \text{ (Eq. wt. of NaOH)}}{10}$

9/29/2021 28

Table-I

Sl. No.	Volume of pipette solution (mL)	Burette reading (mL)		Concordant Value	Indicator
		Initial	Final		
1	**20**	**0**	**19.6**	**19.6**	***Methyl orange***
2	**20**	**0**	**19.6**		

Table-II

Sl. No.	Volume of pipette solution (mL)	Burette Reading (mL)			Concordant Value	
		Initial	Vol. consu. Phenolphthalein end point (A)	Vol. consu. Methyl orange end point (X)	Phenolphthalein	Methyl orange
1	**20**	**0**	**23.9**	**32.8**	**23.9**	**32.8**
2	**20**	**0**	**23.9**	**32.8**		

- Phenolphthalein endpoint (Concordant value) = A = 23.9 mL
- Methyl orange endpoint (Concordant value) = X= 32.8 mL
- Vol. consumed for methyl orange end point after phenolphthalein end point after **B = X-A =32.8 - 23.9 = 8.9 mL**

Result

- Normality of the given HCl solution = ------------
- Amount of Na_2CO_3 present in the given solution = -----
- Amount of NaOH present in the given solution = -----

31

Calculation, standardisation

Volume of Na_2CO_3 solution V_2 = 20 mL

Normality of Na_2CO_3 solution N_2 = 0.05 N (given in bottle)

Volume of HCl solution V_1 = 19.6 mL

Normality of HCl solution N_1 = **<u>0.051 N</u>**

32

Calculation

Estimation of amount of Na_2CO_3

Volume of HCl solution (2B) V_1 = 17.8 mL

Normality of HCl solution N_1 = 0.051 N

Volume of mixture V_2 = 20 mL

Normality of Na_2CO_3 N_2 =

= 17.8/20 × 0.051 = 0.04539 N

Normality of Na_2CO_3 = 0.04539 N

Amount of Na_2CO_3 = **0.2405 g**

Calculation

Estimation of amount of NaOH

Volume of HCl solution V_1 = A-B = (23.9 – 8.9) mL = 15 mL

Normality of HCl solution N_1 = 0.051 N

Volume of mixture V_2 = 20 mL

Normality of NaOH N_2 = 0.03825 N

Amount of NaOH = **0.153 g**

Thank you all for your attention

Information presented here were collected from various sources – textbooks, articles, manuscripts, internet and newsletters. All the researchers and authors of the above mentioned sources are greatly acknowledged.