$\begin{array}{c} CH_{3}COONH_{4} + H_{2}O \\ 1 \end{array}$

 $NH_4OH + CH_3COOH$ (Both are weakly ionized)

 $CH_3 COO^- + NH_4^+$

The H^+ and OH^- are equal in concentration; therefore, the solution of this type of salt is **neutral** in nature.

2.9 Buffers, Buffer Solution, Buffer Systems

An electrolyte solution capable of maintaining its pH value relatively constant when either a small acid or base is added is called as buffer solutions. (Or) Solutions of weak acids and salts of their conjugate bases form **buffer solutions**.

Types of Buffers

- 1. Acidic buffers: A buffer of weak acid and its salt, e.g., CH₃COOH and CH₃COONH₄ having pH always less than 7.
- 2. Basic buffers: A buffer of weak base acid and its salt, e.g., NH₄OH and NH₄Cl having pH always greater than 7.

These are the pairs of related chemical compounds. Also in buffer solutions there is a formation of conjugated acid/base pairs.



Buffering Agent

Buffering agents can be either the weak acid or weak base that would comprise a buffer solution. Buffering agents are usually added to water to form buffer solutions. They are the substances that are responsible for the buffering action produced in these solutions. These agents are added to substances that are to be placed into acidic or basic conditions in order to stabilize the substance.

For example, buffered aspirin has a buffering agent, such as MgO, that will maintain the pH of the aspirin as it passes through

76 Pharmaceutical Drug Analysis

of the solution depends on the salt formed, and may not be pH 7.

The choice of an indicator is determined by the pH of the solution at the equivalence point. At the equivalence point of a titration involving ethanoic acid and sodium hydroxide, the only product is an aqueous solution of the ionic compound sodium ethanoate. It is the ethanoate ions behaving as a base that cause the solution at the end-point to have an alkaline pH.

 $CH_3COOH(aq) + OH^{-}(aq) \implies H_2O(l) + CH_3COO^{-}(aq)$ Indeed, the pH of a solution formed at the equivalence point is important because it influences the choice of acid-base indicator for the titration. Acid-base indicators change colour within characteristic pH ranges.

Choosing Indicators for Different Titrations

(1) Strong Acid and a Strong Base

The (Fig. 2.12) shows the pH curve for adding a strong acid to a strong base and superimposed on it are the pH ranges for methyl orange and phenolphthalein. In this curve neither of the indicator changes colour at the equivalence point. However, the graph is so



Fig. 2.12: pH curve for titrating a strong acid with a strong base.

 $ZnO + H_2SO_4 \longrightarrow ZnSO_4 + H_2O.$

Factor: Each ml of $1 \text{N} \text{H}_2 \text{SO}_4$ is equivalent to 0.04068 g of ZnO.

2. Alkalimetry Titration

(A) Direct Titration

(i) Estimation of Boric Acid

IP Limit: It contains not less than 99% and not more than 100.5% of boric acid (H_3BO_3) with reference to dried substances.

Theory: Boric acid is a weak acid hence can't be titrated directly with dissolving in a solution of glycerin in water. The boric acid reacts with glycerin to form glyceryl boric acid which is strong monobasic acid. The glycerol is slightly acidic in nature, therefore, it is neutralized with dilute alkali using phenolphthalein indicator.

 $H_3BO_3 = NaOH = 1000 ml NaOH$ 61.84 g $H_3BO_3 = 1000 ml NaOH$ 0.0618 = 1 ml M NaOH

 \therefore Each 1 ml of 1 M NaOH 0.0618 g of H₃BO₃.

Procedure: Weight accurately 0.25 g of H_3BO_3 and dissolve it in a mixture of 6.5 ml distilled H_2O and 12.5 ml of glycerin. Titrate the required solution with 0.1N NaOH using phenolphthalein as Indicator. The pink colour is obtained at end point.

Factor: Each 1 ml of or 1 M NaOH is equivalent to 0.0618 g of H_3BO_3

Preparation of 0.1N NaOH

Equivalent wt = $\frac{\text{NaOH}}{\text{Basicity}} = \frac{40}{1} = 40 \text{ g}$

Weigh accurately 40 g of NaOH and dissolve it in 1000 ml distilled water = 1 N

For $0.1 \text{ N} \rightarrow$ Weigh accurately 4 g of NaOH solution and dissolve it 250 ml of distilled water. Make up the volume up to 1000 ml in a volumetric flask.

Standardization: With HCl: Pipette 20 ml of 0.1 N NaOH solution into a conical flask, add 2 drops of phenolphthalein and titrate

 $2 \text{ KI} + \text{H}_2 \text{SO}_4 \longrightarrow \text{K}_2 \text{SO}_4 + 2 \text{HI}$

Potassium iodide Hydrogen iodide

Starch mucilage acts as an indicator which gives blue color with free Iodine.

 $I_2 + 2Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$

Sodium thiosulphate

Sodium tetrathionate

Example: Assay of CuSO₄.5H₂O (Copper sulphate) Assay of CaOCl₂ (Chlorinated lime)

Assay of lodine

. ...

I.P.Limit: Iodine contains not less than 99.5% and not more than equivalent of 100.5% of iodine.

Procedure: Weigh accurately iodine (0.5 g) and dissolve in a solution of potassium iodide (1 g) in H_2O (5 ml). Dilute it with water to 25 ml, acidify with acetic acid (1ml) and titrate with 0.1 N sodium thiosulphate till light yellow color is obtained. At this stage, add starch mucilage and the titration is continued till blue color disappears.

 $I_2 + 2Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$ Sodium thiosulphate Sodium tetrathionate

Starch is added toward the end of titration, when most of iodine is reacted with sodium thiosulphate as starch mucilage forms stable complex with excess of iodine.

4. Titration of Ceric Ammonium Sulphate

This titration involves the use of ceric salt which is a powerful oxidising agent. Ceric salt have intense yellow color. The oxidising power of the ceric salts depends upon the concentration and the type of acid used in the assay.

e.g., Ce^{+4} (IV) in HNO₃ and HClO₄ is stronger oxidising agent (monoprotic)

Ce⁺⁴ (IV) in H₂SO₄ are weaker oxidising agent (diprotic acid)

The fluorides and phosphate have tendency to interfere with concern ions because they form precipitates with them as ceric fluorides and ceric phosphate. The end point of cerimetric titration