JHARKHAND
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# PRACTICAL LAB MANUAL

## LAB MANUAL PHARMACEUTICAL ANALYSIS - i

**(B. Pharm I$^{st}$ Year)**

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# EXPERIMENT NO. 1

## 1. OBJECTIVE

To study the steps involved in volumetric analysis.

## 2. REFERENCES

1. Kamboj P.C., Pharmaceutical Analysis, Volume - I, Third edition, 2014, Vallabh Publications, Pages: 53–133.

2. Mendham J. et.al. Vogel’s Textbook of Quantitative Chemical Analysis, Sixth edition, 2006, Dorling Kindersley (India) Pvt. Ltd, Pages: 33 - 36.

## 3. MATERIALS REQUIRED

Spatula, Funnel, Burette, Conical flask, Burette stand, Dropper, Volumetric pipette, Volumetric flask, Glass rod, Beaker, weighing balance.

## 4. THEORY

**Terms involved in volumetric analysis:**

1. **Titrant**: Solution whose concentration is known.
2. **Titrand**: Solution whose concentration is unknown.
3. **Stoichiometric / End point**: It shows that reaction between titrant and titrand is complete.
4. **Standard solution**: Solution whose exact concentration is known.
5. **Titration**: Reaction between titrand and titrant.

**Steps involved in volumetric analysis:**

1. **Method selection**: For analysis of base (NaOH) → acid is used (HCl): Acid – Base titration.
2. **Sampling**: Small amount of chemical is taken as sample.
3. **Solution preparation**: Using appropriate formula, weight of chemical is calculated, weighed and dissolved in suitable solvent.
4. **Removing interferences**: Calibration, blank titration, parallel determination is done.
5. **Observation**: Volume of Titrant used for end point is observed.
6. **Calculation**: Using equivalent factor, concentration of sample solution is calculated.
7. **Result analysis**: Sample pass or fail as per pharmacopoeial standards

## 5. RESULTS

Steps involved in volumetric analysis were studied.

Signature of teacher:……………………

# EXPERIMENT NO. 2

## 1. OBJECTIVE

To prepare and standardize 100 ml of 0.1 N NaOH solution using Oxalic Acid as primary standard.

## 2. REFERENCES

1. Mendham J. et.al. Vogel's Textbook of Quantitative Chemical Analysis, Sixth edition, 2006, Dorling Kindersley (India) Pvt. Ltd, Pages: 40 – 59.

2. Kamboj P.C., Pharmaceutical Analysis, Volume - I, Third edition, 2014, Vallabh Publications, Pages: 270 – 271.

## 3. MATERIALS REQUIRED

**Apparatus Required:**

Funnel, volumetric flasks, conical flask, volumetric pipette, beaker, burette, burette stand, weighing balance.

**Chemicals Required**:

NaOH, oxalic acid, phenolphthalein solution.

## 4. THEORY

Primary standard is a chemical which has following properties: stable, high purity, non hygroscopic, high molecular weight and give stoichiometric reaction. Eg. oxalic acid, KCl.
Secondary standard is a chemical which has following properties: unstable, low purity, hygroscopic, low molecular weight, poor solubility. Eg. HCl, NaOH.

| | **Property** | **Primary Standard** | **Secondary Standard** |
|---|---|---|---|
| 1. | Purity | High | Low |
| 2. | Stability | Stable | Unstable |
| 3. | Oxidisation by air | No | Yes |
| 4. | Hygroscopic | No | Yes |
| 5. | Molecular weight | High | Low |
| 6. | Solubility | Good | Poor |

## 5. PROCEDURE

### 1. Preparation of NaOH solution

…….. g of NaOH was weighed, dissolved in distilled water & volume made upto 100 ml.

### 2. Preparation of 0.1 N Oxalic acid solution.

……..g of Oxalic Acid was weighed, dissolved in distilled water & volume made upto 100 ml.

**3. Standardization of NaOH solution**

10 ml of Oxalic Acid solution & 1-2 drops of phenolphthalein solution were taken in a conical flask. To that solution NaOH solution was added until the solution became just pink.

The titration was performed 3 times along with 3 blank titrations. Volume of NaOH solution used in blank titration was subtracted from the titration value of NaOH to obtain the volume of NaOH solution used against Oxalic acid.

## 6. OBSERVATIONS

**Table 1: Titration of 0.1 N Oxalic Acid solution Vs NaOH solution**

| S. No | Volume of 0.1 N Oxalic Acid solution (ml) | Burette Reading for NaOH Solution (ml) | | |
|---|---|---|---|---|
| | | Initial Reading | Final Reading | Used volume of NaOH |
| 1. | 10 | | | (a) |
| 2. | 10 | | | (b) |
| 3. | 10 | | | (c) |
| Average volume of NaOH solution used in titration = $\frac{a+b+c}{3}$ = …… **A** | | | | |

**Table 2: Blank Titration for 0.1 N Oxalic Acid solution Vs NaOH solution**

| S. No | Volume of Distilled water (ml) | Burette Reading for NaOH Solution (ml) | | |
|---|---|---|---|---|
| | | Initial Reading | Final Reading | Used volume of NaOH |
| 1. | 10 | | | (x) |
| 2. | 10 | | | (y) |
| 3. | 10 | | | (z) |
| Average volume of NaOH solution used in blank titration = $\frac{x+y+z}{3}$ = …… **B** | | | | |

## 7. CALCULATIONS

**Step 1: Calculations for weighing**

weight of the solute (w) = N*EW*V / 1000

### 1: 0.1 N NaOH solution

N = Normality of NaOH solution = 0.1

EW = Equivalent weight of NaOH = Molecular weight /1 =

V = Volume of NaOH solution = 100 ml

Thus w of NaOH taken = * * / 1000 = ……… g

### 2: 0.1 N Oxalic Acid solution

N = Normality of Oxalic Acid solution = 0.1

EW = Equivalent weight of Oxalic Acid = Molecular weight /2 =

V = Volume of Oxalic Acid solution = 100 ml

Thus w of Oxalic Acid taken = * * / 1000 = ……. g

### Step 2: Calculation for Standardization of NaOH solution

$\mathbf{N}_{\text{NaOH}}\ \mathbf{V}_{\text{NaOH}} = \mathbf{N}_{\text{Oxalic Acid}}\ \mathbf{V}_{\text{Oxalic Acid}}$

Here

$V_{\text{NaOH}}$ = volume used in titration (A) - volume used in blank titration (B) =

$N_{\text{Oxalic Acid}} = 0.1$

$V_{\text{Oxalic Acid}} = 10$

Thus $$N_{\text{NaOH}} = \frac{N_{\text{Oxalic Acid}}\ V_{\text{Oxalic Acid}}}{V_{\text{NaOH}}} = \frac{0.1 \times 10}{\ldots\ldots\ldots} = \ldots\ldots\ \text{N}$$

## 8. RESULTS

NaOH solution was prepared & was found to be …... N.

Signature of teacher:……………………

# EXPERIMENT NO. 3

## 1. OBJECTIVE

To perform the assay of ammonium chloride as per Indian Pharmacopoeia 2014.

## 2. REFERENCE

Indian Pharmacopoeia, Volume II, 2014, Pages: 1049-1050.

## 3. MATERIALS REQUIRED

**Apparatus Required:**

Funnel, volumetric flasks, conical flask, volumetric pipette, graduated pipette, burette, burette stand, rubber bulb, weighing balance.

**Chemicals Required:**

Sample (Ammonium chloride), formaldehyde solution, phenolphthalein solution, 0.1 M sodium hydroxide solution.

## 4. THEORY

"Acid base indicator is a chemical which determine the end point in neutralization titration by changing colour according to pH of titrand solution".

Ostwald theory: Acid base indicator is either a weak acid or a weak base having different colour in ionized and unionized forms.

Example: Phenolphthalein (Hln) is a weak acid. It show pink coloured in basic medium and is colourless in acidic medium.

$$\mathrm{Hln} \rightleftharpoons \mathrm{H^+ + ln^-}$$

Phenolphthalein in basic solution: $OH^-$ combine with $H^+$ of indicator to form $H_2O$. To maintain equilibrium, Hln is ionized (forward reaction increases) to increase the amount of $ln^-$ and indicator is colourless.

Phenolphthalein in acidic solution: HCl decreases the ionization of Hln due to common ion effect. This increases the rate of backward reaction (amount of unionized Hln increases) and indicator show pink colour.

Strong electrolyte: $\mathrm{HCl} \rightleftharpoons \mathrm{H^+ + Cl^-}$
Weak electrolyte: $\mathrm{Hln} \rightleftharpoons \mathrm{H^+ + ln^-}$

## 5. PROCEDURE (I.P. 2014):

Dissolved 0.1 g ammonium chloride in 20 ml of water and added a mixture of 5 ml of formaldehyde solution, previously neutralized to dilute phenolphthalein solution, and 20 ml of water. After 2 minutes, titrated slowly with 0.1 M sodium hydroxide using a further 0.2 ml of phenolphthalein solution as indicator.

## 6. OBSERVATIONS

**Table 1: Titration of ammonium chloride Vs NaOH solution**

| S. No | ammonium chloride (g) | Burette Reading for NaOH Solution (ml) | | |
|---|---|---|---|---|
| | | Initial Reading | Final Reading | Used volume of NaOH |
| 1. | 10 | | | (a) |
| 2. | 10 | | | (b) |
| 3. | 10 | | | (c) |
| Average volume of NaOH solution = $\frac{a+b+c}{3}$ = **A** | | | | |

**Table 2: Blank titration for ammonium chloride Vs NaOH solution**

| . No | Ammonium chloride (g) | Burette Reading for NaOH Solution (ml) | | |
|---|---|---|---|---|
| | | Initial Reading | Final Reading | Used volume of NaOH |
| 1. | 0 | | | (x) |
| 2. | 0 | | | (y) |
| 3. | 0 | | | (z) |
| Average volume of NaOH solution = $\frac{x+y+z}{3}$ = **B** | | | | |

## 7. CALCULATIONS

**Estimation of ammonium chloride**

**Equivalent factor:** 1 ml of 0.1 M NaOH is equivalent to 0.005349 g of $NH_4Cl$.

Here

$V_{NaOH}$ = volume used in titration (A) - volume used in blank titration (B) =………

$M_{NaOH}$ =

Each ml of 0.1 M NaOH is equivalent to 0.005349 g of $NH_4Cl$.

Thus each ml of $M_{NaOH}$ NaOH is equivalent to $M_{NaOH}$ X 0.005349 g of $NH_4Cl$.

Thus $V_{NaOH}$ ml of $M_{NaOH}$ NaOH is equivalent to $V_{NaOH}$ X $M_{NaOH}$ X 0.005349 g of $NH_4Cl$.

= ………. g of $NH_4Cl$.

= Experimental value

Claimed amount of $NH_4Cl$ = quantity of $NH_4Cl$ taken in the experiment = 0.1 g

$$\text{Percentage Purity of ammonium chloride} = \frac{\text{Experimental amount of } NH_4Cl}{\text{Claimed Amount of } NH_4Cl} \times 100$$

$$= \frac{\qquad}{\qquad} \times 100 = \qquad \%$$

## 8. RESULTS

The percentage purity of the given ammonium chloride was found to be ………. %.

Signature of teacher:……………………

# EXPERIMENT NO. 4

## 1. OBJECTIVE

To prepare and standardize 100 ml of 0.05 N sodium thiosulphate solution using Potassium Iodate as primary standard.

## 2. REFERENCES

1.. Mendham J. et.al. Vogel's Textbook of Quantitative Chemical Analysis, Sixth edition, 2006, Dorling Kindersley (India) Pvt. Ltd, Pages: 456-466.

2.. Beckett A.H., Stenlake J.B., Practical Pharmaceutical Chemistry, Fourth edition - Part One, CBS Publishers. Page: 187.

## 3. MATERIALS REQUIRED

### Apparatus Required:

Funnel, volumetric flasks, iodine flask, volumetric pipette, graduated pipette, burette, burette stand, rubber bulb, weighing balance.

**Chemicals Required**:

$Na_2S_2O_3$, $KIO_3$, KI, $H_2SO_4$.

## 4. THEORY

**"Iodometry** is a redox titration in which iodine is formed by reaction of sample with excess of iodide". It is used in for quantitative reduction of oxidising agents. It involves:

HI formation: $2KI + H_2SO_4 \rightarrow HI + K_2SO_4$

Molecular iodine formation: $HI + KIO_3 \rightarrow I_2$

Titration: $I_2$ + standard $Na_2S_2O_3 \rightarrow 2I^-$

In presence of light $I^-$ is oxidised by oxygen to $I_2$. Thus iodometry is done in diffused light / dark.

$$I^- + 4H^+ + O_2 \rightarrow 2I_2 + 2H_2O$$

**Titrand / titrant:** Iodide ($I^-$). **Molecular iodine** is formed. Molecular iodine is highly volatile and sparingly soluble in water and it is retained in the titrand mixture due to formation of potassium tri-iodide with KI. **Indicator:** Iodine itself act as indicator (self indicator). Iodine is pale yellow in colour and iodide is colourless. Starch act as external indicator and iodine forms blue coloured complex with starch. **Iodine flask:** Iodometry is carried in iodine flask to prevent the Oxidation of KI by atmospheric oxidation & Loss of iodine formed due to the chemical reaction. **Oxidizing agent:** Example: $KIO_3$, **Reducing agent:** $I^-$. **Titration:** $I^-$ reacts with $KIO_3$ to form $I_2$.

## 5. PROCEDURE

### 1. Preparation of $Na_2S_2O_3$ solution

………g of $Na_2S_2O_3$ was weighed, dissolved in distilled water & volume made upto 100 ml.

### 2. Standardization of $Na_2S_2O_3$ solution

**a. Preparation of 0.05 N $KIO_3$ solution.**

…g of $KIO_3$ was accurately weighed, dissolved in distilled water & volume made upto 100 ml.

**b. Titration of 0.05 N $KIO_3$ solution Vs $Na_2S_2O_3$ solution**

10 ml of $KIO_3$ solution, 5.2 g of KI & 4 ml of 1 M $H_2SO_4$ were taken in an Iodine flask. Flask was closed, shaken & placed in dark for 10 minutes. To that solution $Na_2S_2O_3$ solution was added until the solution became colourless.

Blank titration can not be performed as in absence of $KIO_3$, iodine will not evolve to give colour to the titrand solution.

## 6. OBSERVATIONS

**Table 1: Titration of $Na_2S_2O_3$ solution Vs 0.05 N $KIO_3$ solution**

| S. No. | Volume of 0.05 N $KIO_3$ solution (ml) | Burette Reading for $Na_2S_2O_3$ solution (ml) | | |
|---|---|---|---|---|
| | | Initial Reading | Final Reading | Used volume of $Na_2S_2O_3$ |
| 1. | 10 | | | (a) |
| 2. | 10 | | | (b) |
| 3. | 10 | | | (c) |
| Average volume of $Na_2S_2O_3$ solution = $\frac{a+b+c}{3}$ = | | | | |

## 7. CALCULATIONS

**Step 1: Calculations for weighing**

weight of solute (g) = N.EW.V / 1000

**1: 0.05 N $Na_2S_2O_3$ solution**

N = Normality of $Na_2S_2O_3$ solution = 0.05

EW = Equivalent weight of $Na_2S_2O_3$ = 158 / 1 = 158

V = Volume of $Na_2S_2O_3$ solution in ml = 100 ml

Thus w of $Na_2S_2O_3$ taken = * * /1000 = …….. g

**2: 0.05 N $KIO_3$ solution**

N = Normality of $KIO_3$ solution = 0.05

EW = Equivalent weight of $KIO_3$ = 214 / 6 = 35.667

V = Volume of $KIO_3$ solution in ml = 100 ml

Thus w of $KIO_3$ taken = * * /1000 = …….. g

### Step 2: Calculation for Standardization of $Na_2S_2O_3$ solution

$N_{Na2S2O3}\ V_{Na2S2O3} = N_{KIO3}\ V_{KIO3}$

Here $N_{KIO3} = 0.05$

$V_{KIO3} = 10$

$V_{Na2S2O3} =$

$$\text{Thus } N_{Na2S2O3} = \frac{N_{KIO3} \times V_{KIO3}}{V_{Na2S2O3}}$$

$=$

## 8. RESULTS

Sodium thiosulphate solution was prepared & found to be____N.

Signature of teacher:……………………

# EXPERIMENT NO. 5

## 1. OBJECTIVE

To prepare and standardize 100 ml of 0.02 N $KMnO_4$ solution using sodium oxalate as primary standard.

## 2. REFERENCES

1. Mendham J. et.al. Vogel's Textbook of Quantitative Chemical Analysis, Sixth edition, 2006, Dorling Kindersley (India) Pvt. Ltd, Pages: 447-450.

2. Beckett A.H., Stenlake J.B., Practical Pharmaceutical Chemistry, Fourth edition - Part One CBS Publishers. Pages: 177 – 178.

## 3. MATERIALS REQUIRED

**Apparatus Required:**

Funnel, volumetric flasks, conical flask, volumetric pipette, graduated pipette, burette, burette stand, rubber bulb, water bath, tripod stand, weighing balance.

**Chemicals Required**:

$KMnO_4$, $Na_2C_2O_4$, sulphuric acid.

## 4. THEORY

Redox titration is based on change in electrode potential of sample during redox reaction. Oxidation is the loss of electrons / increase in oxidation number. Reduction is the gain of electrons / decrease in oxidation number.

Electrode potential is the tendency of element for loss or gain of electrons, when element is in contact with its ion. It is calculated by Nerst equation

$$E = E^\circ + \frac{0.059}{n} \log \frac{[\text{oxidized form}]}{[\text{reduced form}]}$$

E = electrode potential

E° = standard electrode potential

n = number of electrons gained or lost.

Redox curve: is curve between electrode potential of titrand and volume of titrant. Example: Titration of iodine (titrand) and sodium thiosulphate (titrant).

$$Na_2S_2O_3 + I_2 \rightarrow 2I^- + 2Na_2S_4O_6$$

## 5. PROCEDURE

### 1. Preparation of $KMnO_4$ solution

……….. g of $KMnO_4$ was weighed, dissolved in distilled water & volume made upto 100 ml.

### 2. Preparation of 0.02 N $Na_2C_2O_4$ solution

……….. g of $Na_2C_2O_4$ was accurately weighed, dissolved in distilled water & volume made upto 100 ml.

**3. Standardization of $KMnO_4$ solution**

10 ml of $Na_2C_2O_4$ solution, 2.5 ml of 1 M sulphuric acid were warmed upto 60-70°C in a conical flask using water bath. To that solution, $KMnO_4$ solution was added until the solution became just pink.

The titration was performed 3 times along with 3 blank titrations. Volume of the $KMnO_4$ solution used in blank titration was subtracted from the titration value of $KMnO_4$ to obtain the volume of $KMnO_4$ solution used against $H_2O_2$ solution.

## 6. OBSERVATIONS

**Table 1: Titration of 0.02 N $Na_2C_2O_4$ solution Vs $KMnO_4$ solution**

| . S. No | Volume of 0.02 N $Na_2C_2O_4$ solution (ml) | Burette Reading for $KMnO_4$ Solution (ml) | | |
|---|---|---|---|---|
| | | Initial Reading | Final Reading | Used volume of $KMnO_4$ |
| 1. | 10 | | | (a) |
| 2. | 10 | | | (b) |
| 3. | 10 | | | (c) |
| Average volume of $KMnO_4$ solution = $\frac{a+b+c}{3}$ = **A** | | | | |

**Table 2: Blank titration for 0.02 N $Na_2C_2O_4$ solution Vs $KMnO_4$ solution**

| . S. No | Volume of Distilled water (ml) | Burette Reading for $KMnO_4$ Solution (ml) | | |
|---|---|---|---|---|
| | | Initial Reading | Final Reading | Used volume of $KMnO_4$ |
| 1. | 10 | | | (x) |
| 2. | 10 | | | (y) |
| 3. | 10 | | | (z) |
| Average volume of $KMnO_4$ solution = $\frac{x+y+z}{3}$ = **B** | | | | |

## 7. CALCULATIONS

**Step 1: Calculations for weighing**

weight of the solute (w) = N*EW*V / 1000

**1: 0.02 N $Na_2C_2O_4$ solution**

N = Normality of $Na_2C_2O_4$ solution = 0.02

EW = Equivalent weight of $Na_2C_2O_4$ = Molecular weight /2 =

V = Volume of $Na_2C_2O_4$ solution = 100 ml

Thus w of $Na_2C_2O_4$ taken = * * / 1000 = …….. g

**2: 0.02 N $KMnO_4$ solution**

N = Normality of $KMnO_4$ solution = 0.02

EW = Equivalent weight of $KMnO_4$ in acidic medium = Molecular weight /5 =

V = Volume of $KMnO_4$ solution = 100 ml

Thus w of $KMnO_4$ taken = * * / 1000 = ……… g

**Step 2. Calculation for Standardization of $KMnO_4$ solution**

$N_{KMnO4} V_{KMnO4} = N_{Na2C2O4} V_{Na2C2O4}$

Here $V_{KMnO4}$ = volume used in titration (A) - volume used in blank titration (B)

$N_{Na2C2O4} = 0.02$

$V_{Na2C2O4} = 10$

$$\text{Thus} \quad N_{KMnO4} = \frac{N_{Na2C2O4} V_{Na2C2O4}}{V_{KMnO4}}$$

$$= \ldots\ldots N$$

## 8. RESULTS

$KMnO_4$ solution was prepared & found to be ….N.

# EXPERIMENT NO. 6

## 1. OBJECTIVE

To determine the percentage purity of given $H_2O_2$ sample (20 Volume).

## 2. REFERENCES

1. Kushwaha S.P. and Gupta S.K., Pharmaceutical Analysis - I, First edition, 2018, R. Narain Publishers & Distributors, Pages: 140 - 141.

2. Indian Pharmacopoeia 2014, Volume II, Page: 1912.

## 3. MATERIALS REQUIRED

**Apparatus Required:**

Funnel, volumetric flasks, conical flask, volumetric pipette, graduated pipette, burette, burette stand, tile, rubber bulb, water bath, tripod stand, weighing balance.

**Chemicals Required:**

$KMnO_4$, $H_2O_2$ (20 volume), sulphuric acid.

## 4. THEORY

"Redox indicator is a chemical which indicates the end point in redox titration by virtue of different colours in oxidized and reduced forms". $In_{\text{oxidized form}} + ne \rightleftharpoons In_{\text{oxidized form}}$

When n = 1 and

**i.** Electrode potential of titrand is more than ($E°_{ln}$ + 0.06) → colour of indicator is of oxidized form

**ii.** Electrode potential of titrand is less than ($E°_{ln}$ - 0.06) → colour of indicator is of reduced form.

**iii.** Electrode potential of titrand is between ($E°_{ln}$ ± 0.06) → colour of indicator is due to mixture of oxidized and reduced form. This range ($E°_{ln}$ ± 0.06) is known as potential range and during this range, colour of indicator is not clear.

Example: Ferroin: $E°_{ln}$ = 1.14 V

**Types of redox indicators:**

1. Self indicator: Titrant or titrand act as indicator. Eg: $KMnO_4$, $I_2$

$$\underset{\text{pink}}{KMnO_4} \rightarrow \underset{\text{colourless}}{Mn^{++}}$$

2. Internal indicator: is added in the titration vessel (conical flask). eg: Diphenylamine.

3. External indicator: is used outside the titration vessel (conical flask). Titrand is taken out of the conical flask and added to indicator. Eg: Starch (iodine gives blue colour with starch).

## 5. PROCEDURE (I.P. 1996)

1 ml of $H_2O_2$ (20 Volume) solution, 20 ml of 1 M sulphuric acid were taken in a conical flask. To that solution 0.02 M $KMnO_4$ solution was added until the solution became pink.

Titration was performed 3 times along with 3 blank titrations. Volume of the $KMnO_4$ solution used in blank titration was subtracted from the titration value of $KMnO_4$ to obtain the volume of $KMnO_4$ solution used against $H_2O_2$ solution.

## 6. OBSERVATIONS

**Table 1: Titration of $H_2O_2$ solution Vs $KMnO_4$ solution**

| S. No | Volume of $H_2O_2$ solution (ml) | Burette Reading for $KMnO_4$ Solution (ml) | | |
|---|---|---|---|---|
| | | Initial Reading | Final Reading | Used volume of $KMnO_4$ |
| 1. | 01 | | | (a) |
| 2. | 01 | | | (b) |
| 3. | 01 | | | (c) |
| Average volume of $KMnO_4$ solution = $\frac{a+b+c}{3}$ = **A** | | | | |

**Table 2: Blank titration for $H_2O_2$ solution Vs $KMnO_4$ solution**

| S. No | Volume of $H_2O_2$ solution (ml) | Burette Reading for $KMnO_4$ Solution (ml) | | |
|---|---|---|---|---|
| | | Initial Reading | Final Reading | Used volume of $KMnO_4$ |
| 1. | 0 | | | (x) |
| 2. | 0 | | | (y) |
| 3. | 0 | | | (z) |
| Average volume of $KMnO_4$ solution = $\frac{x+y+z}{3}$ = **B** | | | | |

## 7. CALCULATIONS

### Estimation of $H_2O_2$ strength

Here $V_{KMnO4}$ = volume used in titration (A) - volume used in blank titration (B)

=

$N_{KMnO4}$ = ……. $M_{KMnO4}$

Each ml of 0.02 M $KMnO_4$ is equivalent to 0.001701 g of $H_20_2$

Thus each ml of $M_{KMnO4}$ $KMnO_4$ is equivalent to $M_{KMnO4}$ X 0.001701 g of $H_20_2$

Thus $V_{KMnO4}$ ml of $M_{KMnO4}$ $KMnO_4$ is equivalent to $V_{KMnO4}$ X $M_{KMnO4}$ X 0.001701 g of $H_20_2$

= ………. g of $H_20_2$

= Experimental value

Claimed amount of $H_2O_2$ (quantity of $H_2O_2$ taken in the experiment) =

Percentage Purity of ammonium chloride = $\frac{\text{Experimental amount of } H_2O_2}{\text{Claimed Amount of } H_2O_2} \times 100$

= ______ X 100 = …….… %

## 8. RESULTS

The percentage purity of the given $H_2O_2$ sample was found to be ……. %.

## EXPERIMENT NO. 7

### 1. OBJECTIVE

To prepare and standardize 100 ml 0.05 M disodium edetate solution.

### 2. REFERENCE

1. Kushwaha S.P. and Gupta S.K., Pharmaceutical Analysis - I, First edition, 2018, R. Narain Publishers & Distributors, Pages: 118 - 119.

2. Beckett A.H., Stenlake J.B., Practical Pharmaceutical Chemistry, Fourth edition - Part One CBS Publishers. Pages: 219 – 220.

### 3. MATERIALS REQUIRED

**Apparatus Required:**

Funnel, volumetric flasks, conical flask, volumetric pipette, graduated pipette, burette, burette stand, tile, rubber bulb, water bath, tripod stand, weighing balance.

**Chemicals Required**:

Disodium edetate, Mg ribbon, HCl, ammonia buffer solution, Eriochrome Black T, Sodium Chloride.

### 4. THEORY

"Complexometric titration is an analytical method in which volume of complexing agent used to convert sample into complex is measured".

"Complexing agent (ligand) is an ion or molecule which form covalent or coordinate bond with metal ion to form complex". It has O, N, S and donate electrons to metal ions to form coordinate bond.

**Buffer solution**

In complexometric reaction, acid is formed and reaction is reversible.

$$M^{++} + [EDTA]^{--} \rightleftharpoons [MX]^{--} + 2H^{+}$$

Complex of alkaline metal are stable in alkaline pH and decompose in neutral and basic medium.

Alkaline buffer are used to maintain pH 10. Buffer also prevent the backward reaction by neutralizing the acid formed in the titration reaction. Eg: $NH_3$ / $NH_4Cl$ buffer.

### 4. PROCEDURE

**1. Preparation of disodium edetate solution**

……….. g of disodium edetate was weighed, dissolved in distilled water & volume made upto 100 ml.

**2. Standardization of disodium edetate solution**

Weighted exactly about 0.24 g of metallic mg ribbon. dissolved metal in 25 ml of 1m hcl solution and diluted in volumetric flask to 1000 ml.
Transferred 25 ml of magnesium solution to conical flask, added 75 ml of distilled water, 2 ml of pH10 ammonia buffer solution and a pinch of eriochrome black t [1% (w/w) in sodium chloride].
Titrate with edta solution till the color changes to blue.

The titration was performed 3 times along with 3 blank titrations. Volume of the disodium edetate solution used in blank titration was subtracted from the titration value of disodium edetate to obtain the volume of disodium edetate solution used against Mg.

## 6. OBSERVATIONS

### Step 1: Observation Tables

**Table 1: Titration of Zn solution Vs disodium edetate solution**

| S. No | Volume of Zn solution (ml) | Burette Reading for disodium edetate Solution (ml) | | |
|---|---|---|---|---|
| | | Initial Reading | Final Reading | Used volume of EDTA |
| 1. | 10 | | | (a) |
| 2. | 10 | | | (b) |
| 3. | 10 | | | (c) |
| Average volume of disodium edetate solution = $\frac{a+b+c}{3}$ = | | | | **A** |

**Table 2: Blank titration for Zn solution Vs disodium edetate solution**

| S. No | Volume of Distilled water (ml) | Burette Reading for disodium edetate Solution (ml) | | |
|---|---|---|---|---|
| | | Initial Reading | Final Reading | Used volume of EDTA |
| 1. | 10 | | | (x) |
| 2. | 10 | | | (y) |
| 3. | 10 | | | (z) |
| Average volume of disodium edetate solution = $\frac{x+y+z}{3}$ = | | | | **B** |

## 7. CALCULATIONS

### Step 1: Calculations for weighing

weight of the solute (w) = M*MW*V / 1000

**1: 0.05 M disodium edetate solution**

M = Molarity of disodium edetate solution = 0.05

MW = Molecular weight of disodium edetate =

V = Volume of disodium edetate solution = 100 ml

Thus w of disodium edetate taken = * * /1000 = g

**Step 3: Calculation for Standardization of disodium edetate solution**

**Equivalent factor:** 1 ml of 0.1 M EDTA ≡ 0.638 mg of Zn

Here

$V_{\text{disodium edetate}}$ = volume used in titration (A) - volume used in blank titration (B) =

Weight of Zinc in 10 ml = 0.024 g

$$\text{Molarity of EDTA} = \frac{\text{weight of Zn in 10 ml X 1000}}{\text{65.38 X volume of EDTA solution used in titration}}$$

$$= \dots\dots\dots \text{ M}$$

## 8. RESULTS

Disodium edetate solution was prepared & found to be ….M.

# EXPERIMENT NO. 8

## 1. OBJECT

To determine the normality of base by conductometric titration.

## 2. REFERENCES

1. Kushwaha S.P. and Gupta S.K., Pharmaceutical Analysis - I, First edition, 2018, R. Narain Publishers & Distributors, Pages: 156.

2. Nema R.K., et. al., A Practical approach to Pharmaceutical Analysis, First edition, 2008, CBS Publisher, Pages: 116 - 123.

## 3. MATERIALS REQUIRED

**Apparatus Required:**

Funnel, beaker, glass rod, burette, burette stand, rubber bulb, conductometer.

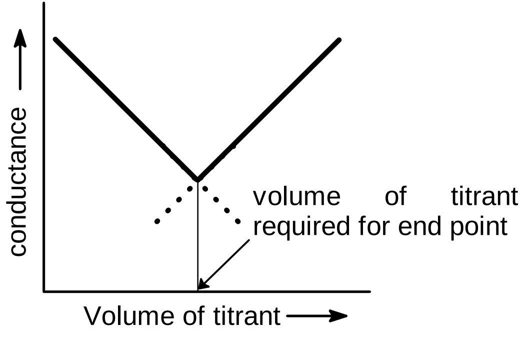
**Chemicals Required:**

HCl sample solution, standard NaOH solution

## 4. THEORY

Conductance is the ability of ions to conduct electricity in an electrolyte solution. It is the reciprocal of resistance. Unit of conductance = 1/ ohm = ohm$^{-1}$ = mho = Siemens

During titration, an ion is substituted by other ion. Conductance is increased or decreased depending on the conductance of latter ion (more or less mobility than earlier ion). Titrant should be 10-20 times more concentrated than titrand to prevent the change in titrand volume.



Conductivity cell contains sample and represents an arm of the Wheatstone bridge. Electrodes are made up of platinum black coated platinum plates (area of 1 cm$^2$) and are kept at a distance of 1 cm from each other. Electrodes are connected through platinum wires to Wheatstone bridge.

## 5. PROCEDURE

1. Filled the standard HCl solution in the burette
2. Took 25 ml of sample solution in a beaker.
3. Dipped the electrode in the sample solution.
4. After adjusting the cell constant, noted the conductance of the sample solution.
5. Added 1 ml of the standard HCl solution from the burette and stirred the sample solution for proper neutralization.
6. Noted the conductance of the sample solution again.

7. Repeatedly added 1 ml of standard HCl solution from the burette and stirred the sample solution to obtain readings.
8. Plotted the graph between conductance (y axis) and volume of standard HCl solution (x axis) added from the burette.

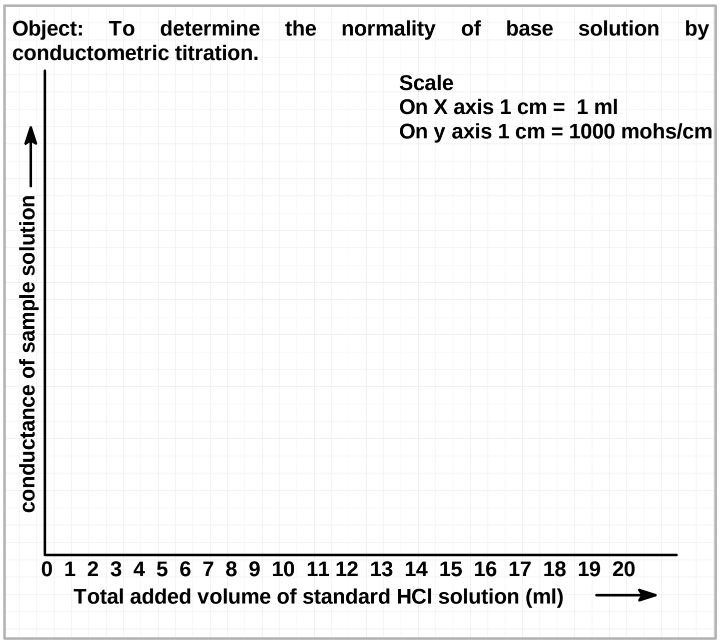
## 6. OBSERVATIONS

### Step 1: Observation Table

**Table 1: Titration of base vs acid by conductometric titration**

| **S. No.** | Volume of standard HCl solution added from the burette (ml) | Total Volume of standard HCl solution added from the burette (ml) | Conductance of sample solution |
|---|---|---|---|
| 1. | 0 | 0 | |
| 2. | 1 | 1 | |
| 3. | 1 | 2 | |
| 4. | 1 | 3 | |
| 5. | 1 | 4 | |
| 6. | 1 | 5 | |
| 7. | 1 | 6 | |
| 8. | 1 | 7 | |
| 9. | 1 | 8 | |
| 10. | 1 | 9 | |
| 11. | 1 | 10 | |

### Step 2: Graphical Representation



**Object: To determine the normality of base solution by conductometric titration.**

**Scale**
**On X axis 1 cm = 1 ml**
**On y axis 1 cm = 1000 mohs/cm**

## 7. CALCULATIONS

**Normality of strong acid solution**

$\mathbf{N}_{HCl}\ \mathbf{V}_{HCl} = \mathbf{N}_{NaOH}\ \mathbf{V}_{NaOH}$

Here

$V_{HCl}$ = volume used in titration = …… ml

$N_{HCl}$ = 1 N (given)

$V_{NaOH}$ = 25 ml

Thus $N_{NaOH} = \dfrac{N_{HCl}\ V_{HCl}}{V_{NaOH}}$ = …….. N

## 8. RESULTS

Normality of base by conductometric titration was found to be ……. N

# EXPERIMENT NO. 9

## 1. OBJECTIVE

To determine the normality of strong base by potentiometric titration.

## 2. REFERENCES

1. Kushwaha S.P. and Gupta S.K., Pharmaceutical Analysis - I, First edition, 2018, R. Narain Publishers & Distributors, Pages: 166 - 167.

2. Nema R.K., et. al., A Practical approach to Pharmaceutical Analysis, First edition, 2008, CBS Publisher, Pages: 76 - 81.

## 3. MATERIALS REQUIRED

**Apparatus Required**:

Funnel, beaker, glass rod, burette, burette stand, rubber bulb, pH meter

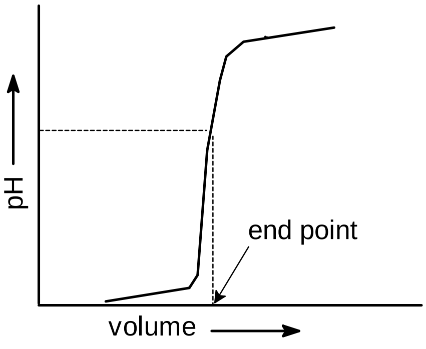
**Chemicals Required**:

NaOH sample solution, standard HCl solution

## 4. THEORY

Glass electrode is used for determination of pH. It allow transfer of H+ through membrane.

Glass electrode has bulb made up of thin glass membrane having $H^+$ ion permeability and high electrical conductivity.



End point determination

Glass electrode develops a potential at the membrane separating the standard HCl and the test solutions due to $H^+$ concentration gradient. Glass bulb function act as semipermeable membrane to $H^+$. Ion exchange reaction takes place between strongly charged cation in glass lattice and proton from the solution.

## 5. PROCEDURE

1. Filled the standard HCl solution in the burette
2. Took 25 ml of sample solution in a beaker.
3. Dipped the electrode in the sample solution.
4. Noted the electrode potential of the sample solution.

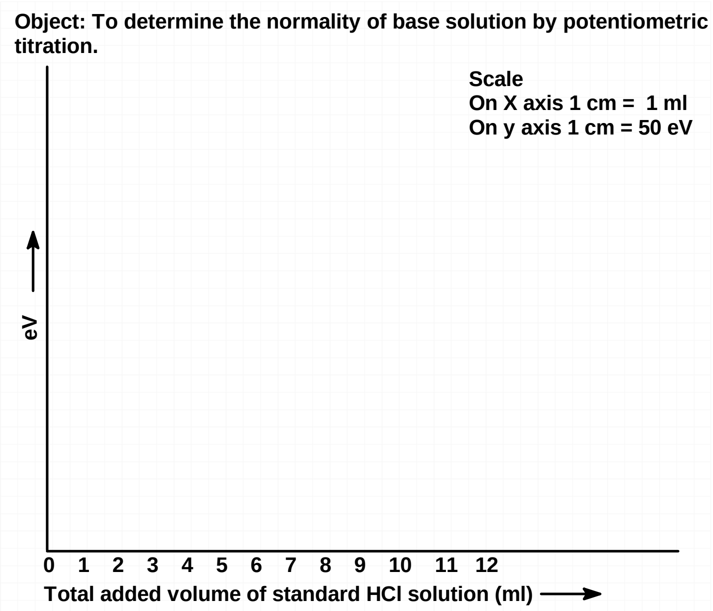
5. Added 1 ml of the standard HCl solution from the burette and stirred the sample solution for proper neutralization.
6. Noted the electrode potential of the sample solution again.
7. Repeatedly added 1 ml of standard HCl solution from the burette and stirred the sample solution to obtain readings.
8. Plotted the graph between electrode potential (y axis) and volume of standard HCl solution (x axis) added from the burette.

## 6. OBSERVATIONS

**Table 1: Titration of base vs acid by potentiometric titration**

| **S. No.** | Volume of standard HCl solution added from the burette (ml) | Total Volume of standard HCl solution added from the burette (ml) | eV |
|---|---|---|---|
| 1. | 0 | 0 | |
| 2. | 1 | 1 | |
| 3. | 1 | 2 | |
| 4. | 1 | 3 | |
| 5. | 1 | 4 | |
| 6. | 1 | 5 | |
| 7. | 1 | 6 | |
| 8. | 1 | 7 | |
| 9. | 1 | 8 | |
| 10. | 1 | 9 | |
| 11. | 1 | 10 | |

### Step 2: Graphical Representation



**Object: To determine the normality of base solution by potentiometric titration.**

**Scale**
**On X axis 1 cm = 1 ml**
**On y axis 1 cm = 50 eV**

## 7. CALCULATIONS

**Normality of strong base solution**

$\mathbf{N}_{NaOH}\ \mathbf{V}_{NaOH} = \mathbf{N}_{HCl}\ \mathbf{V}_{HCl}$

Here

$V_{HCl}$ = volume used in titration = …… ml

$N_{HCl}$ = 1 N (given)

$V_{NaOH}$ = 25 ml

Thus $N_{NaOH} = \dfrac{N_{HCl}\ V_{HCl}}{V_{NaOH}}$ = …….. N

## 8. RESULTS

Normality of base by potentiometric titration was found to be ……. N

# EXPERIMENT NO. 10

## 1. OBJECTIVE

To draw neutralization curve between the given acid-base samples & predict the indicator\s that can be used.

## 2. REFERENCES

1. Kushwaha S.P. and Gupta S.K., Pharmaceutical Analysis - I, First edition, 2018, R. Narain Publishers & Distributors, Pages: 166 - 167.

2. Nema R.K., et. al., A Practical approach to Pharmaceutical Analysis, First edition, 2008, CBS Publisher, Pages: 76 - 81.

## 3. MATERIALS REQUIRED

**Apparatus Required:**

Funnel, beaker, glass rod, burette, burette stand, rubber bulb, pH meter.

**Chemicals Required:**

NaOH solution, HCl solution

## 4. THEORY

"Acid base indicator is a chemical which determine the end point in neutralization titration by changing colour according to pH of titrand solution".

**Ostwald theory**: Acid base indicator is either a weak acid or a weak base having different colour in ionized and unionized forms. Example: **Phenolphthalein** (Hln) is a weak acid. It show pink coloured in basic medium and is colourless in acidic medium.

$$Hln \rightleftharpoons H^+ + ln^-$$

**Phenolphthalein in basic solution**:

$OH^-$ combine with $H^+$ of indicator to form $H_2O$. To maintain equilibrium, Hln is ionized (forward reaction increases) to increase the amount of $ln^-$ and indicator is colourless.

**Phenolphthalein in acidic solution**:

Strong electrolyte: $HCl \rightleftharpoons H^+ + Cl^-$
Weak electrolyte: $Hln \rightleftharpoons H^+ + ln^-$

HCl decreases ionization of Hln due to common ion effect. This increases the rate of backward reaction (amount of unionized Hln increases) and indicator show pink colour.

## 5. PROCEDURE

1. Filled the NaOH solution in the burette
2. Took 25 ml of HCl solution in a beaker.
3. Dipped the electrode in the HCl solution.
4. Noted the pH of the HCl solution.

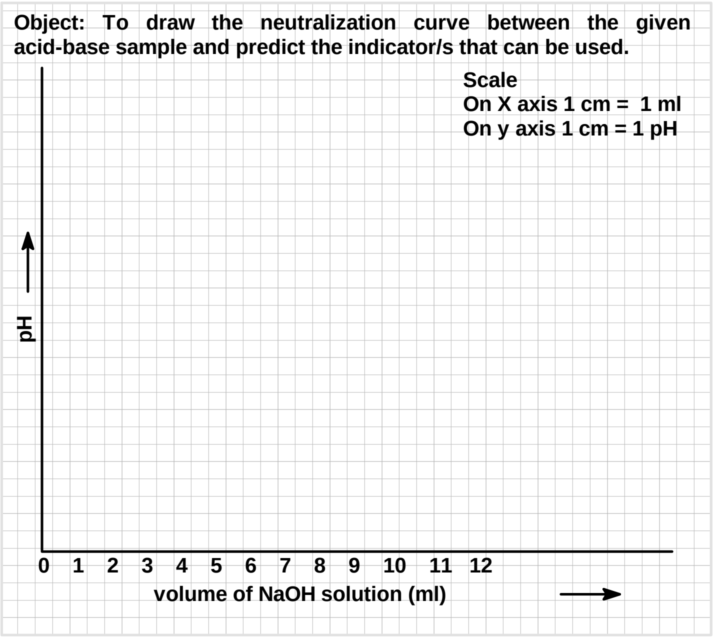
5. Added 1 ml of the NaOH solution from the burette and stirred the HCl solution for proper neutralization.
6. Noted the pH of the HCl solution again.
7. Repeatedly added 1 ml of NaOH solution from the burette and stirred the HCl solution to obtain readings.
8. Plotted the graph between pH (y axis) and volume of NaOH solution (x axis) added from the burette.

## 6. OBSERVATIONS

**Table 1: Titration of base vs acid by pH meter**

| **S. No.** | Volume of NaOH solution added from the burette (ml) | Total Volume of NaOH solution added from the burette (ml) | pH |
|---|---|---|---|
| 1. | 0 | 0 | |
| 2. | 1 | 1 | |
| 3. | 1 | 2 | |
| 4. | 1 | 3 | |
| 5. | 1 | 4 | |
| 6. | 1 | 5 | |
| 7. | 1 | 6 | |
| 8. | 1 | 7 | |
| 9. | 1 | 8 | |
| 10. | 1 | 9 | |
| 11. | 1 | 10 | |

## Step 2: Graphical Representation



**Object: To draw the neutralization curve between the given acid-base sample and predict the indicator/s that can be used.**

**Scale**
**On X axis 1 cm = 1 ml**
**On y axis 1 cm = 1 pH**

## 7. RESULTS

The neutralization curve between the given acid-base samples was drawn & the indicator\s that can be used is\are…………………………………....

# GLOSSARY

- **Sample**: Small amount of pharmaceutical substance representing the property of complete pharmaceutical substance.
- **Analyte**: The component of interest in the sample.
- **Impurity**: A component present in the pharmaceutical product that is not the chemical entity defined as the drug substance or an excipient in the drug product.
- **Pharmaceutical analysis**: The identification and chemical composition determination of the analyte present in the sample of the pharmaceutical substances.
- **Quantitative analysis**: It determine the amount of analyte and impurity present in the sample.
- **Molarity** (M): It indicates the number of moles of solute present per litre of solution". Its unit is moles/litre.
- **1 mole**: Total of the atomic weights of the elements present in the solute. Example: 1 mole NaOH = 23 +16 + 1 = 40g.
- **Molar solution** (1M): It contains 1 mole of solute present in 1 litre of solution. Example: 1 M NaOH solution contains 40 g NaOH in 1 litre of solution.
- **Normality** (N): It indicates the number of gram equivalents of solute present per litre of solution". Its unit is gm equivalents/litre.
- **Volumetric titration**: Reagent solution is added to sample solution (or vice versa) until reaction is complete. Volume of reagent used to achieve end point of the chemical reaction is measured and used for calculating the amount of analyte present in the sample.
- **Titration**: Reaction between titrand and titrant.
- **Standard solution**: It is a solution whose exact concentration is known. It is known as titrant during titration.
- **Titrand**: It is a solution used in titration whose exact concentration is unknown.
- **Standardisation**: It is the process of determining the exact concentration of a solution.
- **Stoichiometric point** / End point / equivalence point: It is the titration point at which chemical reaction between titrant and titrand is complete and titrant and titrand are in equilibrium. Eg: At end point in acid – base titration, moles of $H^+$ are equal to moles of $OH^-$
- **Indicator**: is a chemical which indicates the end point of the titration.
- **Acid base (Neutralization) titration**: Sample / reagent are complimenting acid and base and undergo neutralization reaction with each other.

- **Oxidation-reduction (Redox) titration**: Sample / reagent are complimenting oxidizing agent and reducing agent and undergo redox reaction with each other.

- **Complexometric Titration**: Sample and reagent react to form a complex.

$$CaCO_3 + EDTA \rightarrow Ca\text{-}Edetate$$

- **Electrochemical methods of analysis**: They determine the end point in a titration by measuring the change in electrical property of the sample during the chemical reaction.

- **Conductometry:** It measure the change in conductance due to substitution of one ion by other ion.

- **Potentiometry:** It measure change in electrode potential of sample during redox reaction.

- **Redox reaction:** It is a reaction in which oxidation and reduction takes place together".

$$\underset{\substack{\text{Reduction}\\\text{Oxidizing agent}}}{I_2} \quad + \quad \underset{\substack{\text{oxidation}\\\text{reducing agent}}}{2Cl^-} \rightarrow 2I^- + Cl_2$$

- **Oxidizing agent** (oxidant): It help in oxidation of other chemical and itself gets reduced. Eg. Ceric sulphate, $I_2$, $KIO_3$.

- **Reducing agent** (reductant): It help in the reduction of other chemical and itself gets oxidized. Eg: KI, $Na_2S_2O_3$.

- **Neutralization curve**: It is a curve between pH of titrand (x axis) and volume of titrant used in acid base titration (y axis).