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## VOLUMETRY

## CHEMISTRY PRACTICAL - CLASS: 11; VOLUMETRIC ANALYSIS

## Instructions:

1. After the completion of every titration work, the detailed calculation and result should be attested (before the next practical session) by the teacher-in-charge.
2. You should compulsorily use logarithm table for calculation work.
3. Show detailed calculation work at the side of the work sheet itself.
4. Do not "round off" the results obtained.
5. It is compulsory that completed Journal is submitted for correction in the following week's practical session.
6. In your JOURNAL: Draw all tables and show all calculations (not detailed) on the left hand side and write the rest on the right hand side. (Result should be written on both the sides)
7. INDEX (Journal) may be written in the following format:
8. Main Title: VOLUMETRIC ANALYSIS
9. sub-title: Oxalic acid vs. $\mathbf{N a O H}$; Estimation of $\mathbf{N a O H}$

## PREPARATION OF STANDARD SOLUTION

Expt No:
Date: $\qquad$
Aim: To prepare a 0.1 N solution of oxalic acid.
Requirements: 100 ml volumetric flask, oxalic acid crystals (AR Quality), Weighing bottle, Funnel, Glass rod, wash bottle, balance etc
Theory: A solution whose concentration is known exactly is called a standard solution.
Normality may be defined as the number gram equivalents of solute per liter of the solution and is represented by " N "

Normality $=\frac{\text { Mass of solute } \times 1000}{\text { Equivalent mass } \times \text { volume of solution (ml) }}$

## Equivalent mass of oxalic acid dihydrate $\left(\mathbf{H}_{\mathbf{2}} \mathrm{C}_{\mathbf{2}} \mathbf{O}_{\mathbf{4}} \cdot \mathbf{2} \mathbf{H}_{\mathbf{2}} \mathrm{O}\right)$

RMM of oxalic acid dihydrate $=126 \mathrm{amu}$; and number of replaceable hydrogen in oxalic acid $=2$ Therefore equivalent mass of oxalic acid dihydrate $=126 / 2=63 \mathrm{amu}$.

## Note: Normality of 1 L of solution, which contains 63 g of oxalic acid dihydrate $=1 \mathrm{~N}$

Procedure: Weigh $\mathbf{0 . 6 3 0 0} \mathrm{g}$ of Oxalic acid crystals (AR grade), accurately in a weighing bottle and transfer into a clean funnel placed over a $\mathbf{1 0 0} \mathbf{~ m l}$. standard volumetric measuring flask. Wash down the crystals carefully into the flask by a jet of distilled water. Wash the funnel also down into the flask. Dissolve the crystals completely and then make up the solution to 100 ml . mark. Shake well to make a homogeneous solution.

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## Calculation:

Mass of oxalic acid crystals $\quad=X g=\ldots \ldots \ldots \ldots \ldots \ldots \ldots .$.
Volume of oxalic acid solution prepared $=V m l=100 \mathrm{ml}$.
Normality of the oxalic acid solution $=\mathrm{N}=\frac{X g \times 1000}{63 \times V m l}=\frac{\ldots \ldots \ldots \ldots . . \ldots . . \times 1000}{63 \times 100}=$
Result: The normality of the oxalic acid solution = ....................

## VOLUMETRIC ANALYSIS - I

## ESTIMATION OF SODIUM HYDROXIDE

## Experiment Number: <br> $\qquad$ <br> Date: <br> $\qquad$

Aim: To estimate the strength of a give solution of sodium hydroxide solution by titrating it against a standard solution of oxalic acid.

Requirements: Burette, Pipette, Conical flask, weighing bottle, oxalic acid crystals (A R. Grade) Balance, Funnel, Glass rod, wash bottle etc.

## Indicator used:Phenolphthalein

Theory: The estimation is based on the reaction between oxalic acid and sodium hydroxide as per the following chemical equation

$$
\mathrm{H}_{2} \mathrm{C}_{2} \mathrm{O}_{4}+2 \mathrm{NaOH} \rightarrow \mathrm{Na}_{2} \mathrm{C}_{2} \mathrm{O}_{4}+2 \mathrm{H}_{2} \mathrm{O}
$$

Normality of the given solution is calculated using the law of equivalence formula.

$$
\mathbf{N}_{\mathrm{a}} \mathbf{V}_{\mathrm{a}}=\mathbf{N}_{\mathrm{b}} \mathbf{V}_{\mathbf{b}} \quad \begin{aligned}
& \text { Where, } \\
& \mathrm{N}_{\mathrm{a}}=\text { Normality of the acid; } \quad \mathrm{N}_{\mathrm{b}}=\text { Normality of the base }
\end{aligned}
$$

$\mathrm{V}_{\mathrm{a}}=$ Volume of the acid used; $\mathrm{V}_{\mathrm{b}}=$ Volume of the base used.

## Procedure: Preparation of oxalic acid solution (standard 0.1N)

Weigh $\mathbf{0 . 6 3 0 0} \mathrm{g}$ of Oxalic acid crystals (A R. grade), accurately in a weighing bottle and transfer into a clean funnel placed over a $\mathbf{1 0 0} \mathbf{~ m l}$. standard volumetric measuring flask. Wash down the crystals carefully into the flask by a jet of distilled water. Wash the funnel also down into the flask. Dissolve the crystals completely and then make up the solution to 100 ml . mark. Shake well to make a homogeneous solution

Mass of oxalic acid crystals $\quad=X g=\ldots \ldots \ldots \ldots . . \mathrm{g}$
Volume of solution prepared $\quad=V m l=100 \mathrm{ml}$.
Normality of the oxalic acid solution $=\mathrm{N}=\frac{X g \times 1000}{63 \times \operatorname{Vml}}=\frac{\ldots \ldots \ldots \ldots \ldots \ldots \times 1000}{63 \times 100}=$

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## Estimation of Sodium Hydroxide:

1. Pipette out 20 ml (Wash the pipette thoroughly with distilled water followed by rinsing with $\mathbf{N a O H}$ solution) of the given NaOH into a clean conical flask. (Wash the conical flask thoroughly with distilled water only)
2. Add one drop of phenolphthalein indicator to the flask.
3. Titrate this pink coloured solution against standard oxalic acid solution taken in a clean burette. (Wash the burette thoroughly with distilled water followed by rinsing with oxalic acid solution)
4. End point is marked by the disappearance of the pink colour.
5. End point of the titration is recorded as titre value in a tabular form.
6. Repeat the titration till concordant values are obtained. [Record only two titre (concordant) readings in the table]
7. Using the titre value concentration of the given NaOH is calculated.

|  |  | Burette reading |  | Titre value "Va" <br> Sl No |
| :---: | :---: | :---: | :---: | :---: |
| Volume of <br> NaOH (ml) | Initial reading <br> Y ml | Final reading <br> X ml | (acid consumed) <br> $(\mathbf{X}-\mathbf{Y}) \mathbf{m l}$ |  |
| 1 | 20 ml |  |  |  |
| 2 | 20 ml |  |  |  |

## Calculation:

Volume of NaOH used $\quad=\mathrm{V}_{\mathrm{b}}=20 \mathrm{ml}$

Volume of Oxalic acid used $\quad=\mathrm{V}_{\mathrm{a}} \quad=\ldots \ldots \ldots \ldots \ldots \mathrm{ml}$
Normality of oxalic acid $\quad=\mathrm{N}_{\mathrm{a}} \quad=\ldots \ldots \ldots \ldots \ldots \ldots$

Normality of NaOH

$$
=\mathrm{N}_{\mathrm{b}} \quad=\frac{N_{a} V_{a}}{V_{b}}=\frac{\ldots \ldots \ldots . . \ldots \ldots \ldots \ldots \ldots .}{20}=
$$

Concentration of $\mathrm{NaOH}=$ Normality $\mathbf{x}$ Equivalent mass $=\mathrm{N}_{\mathrm{b}} \mathbf{x} 40=$ $\qquad$ $\mathrm{x} 40=$ $\qquad$ .g/L

## Result:

1. Normality of $\mathbf{N a O H}$
$=$ N
2. Concentration of $\mathbf{N a O H}=$ $\qquad$ .g / L

## VOLUMETRIC ANALYSIS - II

## ESTIMATION OF POTASSIUM HYDROXIDE

## Experiment Number:

$\qquad$
Date:
Aim: To estimate the strength of a give solution of potassium hydroxide solution by titrating it against a standard solution of oxalic acid.

Requirements: Burette, Pipette, Conical flask, weighing bottle, oxalic acid crystals (AR Grade) Balance, Funnel, Glass rod, wash bottle etc.

## Indicator used:Phenolphthalein

Theory: The estimation is based on the reaction between oxalic acid and potassium hydroxide as per the following chemical equation

$$
\mathrm{H}_{2} \mathrm{C}_{2} \mathrm{O}_{4}+2 \mathrm{KOH} \rightarrow \mathrm{~K}_{2} \mathrm{C}_{2} \mathrm{O}_{4}+2 \mathrm{H}_{2} \mathrm{O}
$$

Normality of the given solution is calculated using the law of equivalence formula.

$$
\mathbf{N}_{\mathbf{a}} \mathbf{V}_{\mathrm{a}}=\mathbf{N}_{\mathrm{b}} \mathbf{V}_{\mathrm{b}}
$$

Where, $\mathrm{N}_{\mathrm{a}}=$ Normality of the acid;
$\mathrm{V}_{\mathrm{a}}=$ Volume of the acid used;

$$
\begin{aligned}
& \mathrm{N}_{\mathrm{b}}=\text { Normality of the base } \\
& \mathrm{V}_{\mathrm{b}}=\text { Volume of the base used. }
\end{aligned}
$$

## Procedure:

## Preparation of oxalic acid solution (standard 0.1N)

Weigh 0.6300 g of Oxalic acid crystals (AR. grade), accurately in a weighing bottle and transfer into a clean funnel placed over a $\mathbf{1 0 0} \mathbf{~ m l}$. standard volumetric measuring flask. Wash down the crystals carefully into the flask by a jet of distilled water. Wash the funnel also down into the flask. Dissolve the crystals completely and then make up the solution to 100 ml . mark. Shake well to make a homogeneous solution

Mass of oxalic acid crystals
Volume of solution prepared

Normality of the oxalic acid solution

$$
=X g=\ldots \ldots \ldots \ldots . . g
$$

$$
=V m l=100 \mathrm{ml} .
$$

$$
=\mathrm{N}=\frac{X g \times 1000}{63 \times V m l}=\frac{\ldots \ldots \ldots \ldots . . . . . . . . \times 1000}{63 \times 100}=
$$

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## Estimation of Potassium Hydroxide:

1. Pipette out 20 ml (Wash the pipette thoroughly with distilled water followed by rinsing with KOH solution) of the given KOH into a clean conical flask. (Wash the conical flask thoroughly with distilled water only)
2. Add one drop of phenolphthalein indicator to the flask.
3. Titrate this pink coloured solution against standard oxalic acid solution taken in a clean burette. (Wash the burette thoroughly with distilled water followed by rinsing with oxalic acid solution)
4. End point is marked by the disappearance of the pink colour.
5. End point of the titration is recorded as titre value in a tabular form.
6. Repeat the titration till concordant values are obtained. [Record only two titre (concordant) readings in the table]
7. Using the titre value concentration of the given NaOH is calculated.

|  |  | Burette reading |  | Titre value "Va" <br> SI No |
| :---: | :---: | :---: | :---: | :---: |
| Volume of <br> KOH (ml) | Initial reading <br> Y ml | Final reading of Oxalic <br> X ml | acid consumed) <br> (X $-\mathbf{Y}) \mathbf{~ m l ~}$ |  |
|  |  | 20 ml |  |  |
| 2 | 20 ml |  |  |  |

## Calculation:

| Volume of KOH used | $=\mathrm{V}_{\mathrm{b}}=20 \mathrm{ml}$ |
| :--- | :--- |
| Volume of Oxalic acid used | $=\mathrm{V}_{\mathrm{a}}=\ldots \ldots \ldots \ldots \ldots \mathrm{ml}$ |
| Normality of oxalic acid | $=\mathrm{N}_{\mathrm{a}}=\ldots \ldots \ldots \ldots \ldots . \mathrm{N}$ |
| Normality of KOH | $=\mathrm{N}_{\mathrm{b}}=\frac{N_{a} V_{a}}{V_{b}}=\frac{\ldots \ldots \ldots . \ldots \ldots \ldots \ldots}{20}=\ldots \ldots \ldots \ldots \mathrm{N}$ |

Concentration of $\mathrm{KOH}=$ Normality $\times$ Equivalent mass $=\mathrm{N}_{\mathrm{b}} \times 56=$ $\qquad$ x $56=$ $\qquad$ g/L

## Result:

1. Normality of KOH = .................. N
2. Concentration of $\mathrm{KOH}=$ $\qquad$ .g/L

## VOLUMETRIC ANALYSIS - III

## ESTIMATION OF HYDROCHLORIC ACID

## Experiment Number:

Date: $\qquad$
Aim: To estimate the strength of a give solution of hydrochloric acid solution by titrating it against a standard solution of sodium carbonate.

Requirements: Burette, Pipette, Conical flask, weighing bottle, sodium carbonate crystals (AR Grade) Balance, Funnel, Glass rod, wash bottle etc.

Theory: The estimation is based on the reaction between $\mathrm{HCland} \mathrm{Na}_{2} \mathrm{CO}_{3}$ as per the following chemical equation

$$
2 \mathrm{HCl}+\mathrm{Na}_{2} \mathrm{CO}_{3} \rightarrow 2 \mathrm{NaCl}+\mathrm{H}_{2} \mathrm{O}+\mathrm{CO}_{2}
$$

Normality of the given solution is calculated using the law of equivalence formula.

| $\mathbf{N}_{\mathbf{a}} \mathbf{V}_{\mathbf{a}}=\mathbf{N}_{\mathbf{b}} \mathbf{V}_{\mathbf{b}}$ | Where, <br> $\mathrm{N}_{\mathrm{a}}=$ Normality of the acid; <br> $\mathrm{V}_{\mathrm{a}}=$ Volume of the acid used; | $\mathrm{N}_{\mathrm{b}}=$ Normality of the base <br> $\mathrm{V}_{\mathrm{b}}=$ Volume of the base used. |
| :--- | :--- | :--- |

Indicator used: Methyl Orange

## Procedure: Preparation of $\mathrm{Na}_{2} \underline{C O}_{3}$ solution (standard 0.1 N )

Weigh $\mathbf{0 . 5 3 0 0}$ gof $\mathrm{Na}_{2} \mathrm{CO}_{3}$ crystals (AR grade), accurately in a weighing bottle and transfer into a clean funnel placed over a $\mathbf{1 0 0 . 0} \mathbf{~ m l}$. standard volumetric measuring flask. Wash down the crystals carefully into the flask by a jet of distilled water. Wash the funnel also down into the flask. Dissolve the crystals completely and then make up the solution to 100 ml . mark. Shake well to make a homogeneous solution

Mass of $\mathrm{Na}_{2} \mathrm{CO}_{3}$ crystals $\quad=X g=\ldots \ldots \ldots \ldots . \mathrm{g}$
Volume of solution prepared $\quad=V m l=100 \mathrm{ml}$.
Normality of the $\mathrm{Na}_{2} \mathrm{CO}_{3}$ solution $=\mathrm{N}=\frac{\mathrm{Xg} \times 1000}{53 \times \mathrm{Vml}}=\frac{\ldots \ldots \ldots \ldots \ldots \ldots \times 1000}{53 \times 100}=$ .N

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## Estimation of Hydrochloric acid:

1. Pipette out 20 ml (Wash the pipette thoroughly with distilled water followed by rinsing with $\mathbf{N a}_{2} \mathbf{C O}_{3}$ solution) of the given $\mathrm{Na}_{2} \mathrm{CO}_{3}$ into a clean conical flask (Wash thoroughly with distilled water only).
2. Add a drop of Methyl Orange indicator to the flask.
3. Titrate this yellow coloured solution against hydrochloric acid solution taken in a clean burette. (Wash the burette thoroughly with distilled water followed by rinsing with $\mathbf{H C l}$ acid solution)
4. At the end point the colour of the solution changes from yellow to pink.
5. End point of the titration is recorded as titre value in a tabular form.
6. Repeat the titration till concordant values are obtained. [Record only two titre (concordant) readings in the table]
7. Using the titre value concentration of the given HCl is calculated.

| Sl No | $\begin{gathered} \text { Volume of } \\ \mathrm{Na}_{2} \mathrm{CO}_{3}(\mathrm{ml}) \end{gathered}$ | Burette reading |  | Titre value "Va" (Volume of HCl acid consumed) ( $\mathbf{X}-\mathbf{Y}$ ) ml |
| :---: | :---: | :---: | :---: | :---: |
|  |  | Initial reading Y ml | Final reading X ml |  |
| 1 | 20 ml |  |  |  |
| 2 | 20 ml |  |  |  |

## Calculation:

Volume of $\mathrm{Na}_{2} \mathrm{CO}_{3}$ used $\quad=\mathrm{V}_{\mathrm{b}}=20 \mathrm{ml}$
Volume of HCl acid used $\quad=\mathrm{V}_{\mathrm{a}} \quad=\ldots \ldots \ldots \ldots \ldots \mathrm{ml}$
Normality of $\mathrm{Na}_{2} \mathrm{CO}_{3} \quad=\mathrm{N}_{\mathrm{b}} \quad=\ldots \ldots \ldots \ldots \ldots . . \mathrm{N}$

Normality of HCl

$$
=\mathrm{N}_{\mathrm{a}}=\frac{N_{b} V_{b}}{V_{a}}=\frac{\ldots \ldots \ldots . . \times 20}{\ldots \ldots \ldots \ldots \ldots . . . . . . . . . . . . . . . . . . . ~}
$$

$\qquad$

Concentration of $\mathrm{HCl}=$ Normality $\times$ Equivalent mass $=\mathrm{N}_{\mathrm{a}} \times 36.5=$ $\qquad$ x $36.5=$ $\qquad$ g/L

```
Result:
    1. Normality of HCl = ................ N
2. Concentration of }\mathbf{HCl}```

