CHEMISTRY LABORATORY

NAME OF THE EXPERIMENT — **STANDARDIZATION OF SODIUM HYDROXIDE SOLUTION WITH STANDARD OXALIC ACID SOLUTION.**

Theory

Oxalic acid is a primary and sodium hydroxide is a secondary standard substance. Sodium hydroxide is standardized by titrating it against standard Oxalic acid solution using Phenolphthalein indicator. The reaction between NaOH and Oxalic acid is as follows:

$$2NaOH\,(aq) + \begin{array}{c} COOH \\ | \\ COOH \end{array}.2H_2O\,(aq) \longrightarrow \begin{array}{c} COONa \\ | \\ COONa \end{array}.2H_2O\,(aq) + 2H_2O \qquad (1)$$

Here, by measuring the volume of the NaOH solution required to react completely with a solution of a weighed sample of Oxalic acid the exact concentration of NaOH solution is determined.

Apparatus

- Conical flask
- Pipette
- Burette
- Beaker
- Funnel
- Volumetric flask
- Electric balance

Reagents and Chemicals

- Sodium hydroxide
- Oxalic acid

Indicator

- Phenolphthalein

Procedure

Step-1

Preparation of 100mL 0.05 M Oxalic Acid solution

Molecular weight of Oxalic Acid ($C_2H_2O_4.2H_2O$) = $12\times2 + 1\times2 + 16\times4 + 18\times2 = 126$

1000 mL 1 M oxalic acid contain 126 g oxalic acid

$\therefore$ 100 mL 0.05 M oxalic acid contain $\dfrac{126 \times 100 \times 0.05}{1000}$

$= 0.63$ g oxalic acid

Amount of oxalic acid taken = ... g

$$\text{Strength of the prepared acid solution} = \frac{\text{Weight taken (g)}}{\text{Weight to be taken}} \times 0.05M$$

$$= \frac{\quad}{0.63\text{ g}} \times 0.05M$$

$= \ldots\ldots\ldots\ldots$ M

Transfer . . . . . . g of pure Oxalic Acid in a 100mL volumetric flask, then dissolve it with distilled water and make it upto the mark.

Step-2

Standardization of supplied NaOH solution by the prepared Oxalic Acid solution

1. Take10 mL of supplied oxalic acid solution in a conical flask by means of a pipette.
2. Add 1-3 drop of Phenolphthalein indicator to the solution.
3. Then add NaOH solution drop by drop from a burette. Shake the flask frequently while adding the acid solution. Stop the addition of oxalic acid solution as soon as the **pink color** of the solution just appears.
4. Note the burette reading. The burette reading should be taken carefully at the lower meniscus of the liquid. Difference between initial and final burette reading gives the volume of the NaOH added.
5. Repeat the steps 1-4 at least three times.
6. Calculate the strength of the supplied NaOH solution.

Experimental Data

Table: Standardization of supplied NaOH solution by the prepared Oxalic Acid solution.

No. of observation	Volume of Oxalic acid solution, V_{acid} (mL)	Burette reading (Volume of NaOH)			
		Initial (mL)	Final (mL)	Difference (mL)	Mean V_{base} (mL)
1					
2					
3					

Calculation

Using equation (1) we get, $\frac{V_{acid} \times S_{acid}}{V_{base} \times S_{base}} = \frac{1}{2}$

Here, V_{acid} = ... mL S_{acid} = ... M

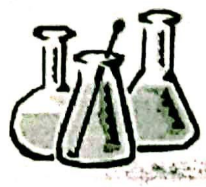
V_{base} = ... mL

∴ S_{base}=............... M

Result

The strength of the supplied NaOH solution is calculated as .... M.

Grade/Marks: _________ Remark:



Signature & Date

Practical Related Questions & Answers

Question: *What is Primary standard substance?*

Answer: The chemical substance that found in pure state and it is possible to weigh accurately in a chemical balance to make a standard solution and the strength of the solution remain unchanged for a long time is called Primary standard substance. Examples: Na_2CO_3, Oxalic acid, $K_2Cr_2O_7$ etc.

Question: *What is Secondary standard substance?*

Answer: The chemical substances that are not found in pure state and it is not possible to weigh accurately in a chemical balance to make a standard solution and the strength of the solution changed for a long time is called secondary standard substance. Examples: NaOH, HCl, $KMnO_4$ etc.

Question: *Why $K_2Cr_2O_7$ is a primary standard substance?*

Answer: $K_2Cr_2O_7$ is a primary standard substance because it has the following characteristics:

1. It is obtained dry and pure state.
2. It is not hygroscopic.
3. It is not oxidized by air and not affected by CO_2.
4. The concentration of the solution produced by $K_2Cr_2O_7$ is unchanged during storage.

Question: *Why $KMnO_4$ is not a primary standard substance?*

Answer: $KMnO_4$ is not a primary standard substance, it is secondary standard substance. Because, it is difficult to obtain the substance ($KMnO_4$) perfectly pure and completely free from manganese dioxide.

Question: *What is Standard solution?*

Answer: The solution of accurately known strength is called the standard solution.

Question: *What is Titration?*

Answer: Titration is a common laboratory method of quantitative chemical analysis that is used to determine the unknown concentration of an identified analyte. Or,

The process, operation, or method of determining the concentration of a substance in solution by adding to it a standard reagent of known concentration in carefully measured amounts until a reaction of definite and known proportion is completed, as shown by a color change or by electrical measurement, and then calculating the unknown concentration.

Question: *What is titrant and titrate?*

Answer: The reagent of known concentration is called the titrant and the substance being titrated is termed titrate.

Question: *Define indicator.*

Answer: Indicator is a substance used in titrations to indicate the completion of a chemical reaction, usually by a change of color.

Question: *Calculate the amount of sodium carbonate in order to prepare 250 ml 0.1(M) solution.*

Answer: Molecular weight of $Na_2CO_3 = 23\times2 + 12 + 16\times3 = 106$

1000ml 1M Na_2CO_3 solutions contains 106 gm Na_2CO_3

$\therefore$ 250 ml 0.1 M Na_2CO_3 solution contains $\dfrac{106 \times 250 \times 0.1}{1000}$

$= 2.65$ gm Na_2CO_3

Question: *Write down the name of indicator used in standardization of HCl with standard Na_2CO_3 solution experiment.*

Answer: Methyl orange.

Question: *What type of standard are Sodium carbonate and Hydrochloric acid?*

Answer: Sodium carbonate (Na_2CO_3) is a Primary standard and Hydrochloric acid (HCl) is a Secondary standard substance.

Question: *20 ml of an HCl solution is required for neutralization of 10 ml 0.1(M) Na_2CO_3 solution. Calculate the strength of the HCl acid.*

Answer: The reaction between Na_2CO_3 and HCl is:

$$Na_2CO_3 + 2HCl = 2NaCl + H_2O + CO_2$$

We have, $$\frac{V_{acid} \times S_{acid}}{V_{base} \times S_{base}} = \frac{2}{1}$$

$V_{base} = 10$ ml $\quad$ $S_{base} = 0.1$ M

$V_{acid} = 20$ ml $\quad$ $S_{acid} = ?$

Putting these values in the above expression, we get,

$\therefore S_{acid} = 0.1$ M

Question: *Calculate the amount of oxalic acid $(COOH)_2.2H_2O$ in order to prepare 250 ml 0.1(M) solution.*

Answer: Molecular weight of Oxalic Acid $(C_2H_2O_4.2H_2O)$ = 12×2 +1×2 + 16×4 + 18×2 = 126

1000 ml 1 M oxalic acid contain 126 gm oxalic acid

∴ 250 ml 0.1 M oxalic acid contain $\dfrac{126 \times 250 \times 0.1}{1000}$

= 3.15 gm oxalic acid

Question: *Write down the name of indicator used in standardization of NaOH with standard oxalic acid $[(COOH)_2.2H_2O]$ solution experiment.*

Answer: Phenolphthalein.

Question: *What type of standards are sodium hydroxide and oxalic acid?*

Answer: Sodium hydroxide (NaOH) is a Secondary standard and Oxalic acid $[(COOH)_2.2H_2O]$ is a Primary standard substance.

Question: *20 ml of a NaOH solution is required for neutralization of 25 ml 0.1(M) oxalic acid solution. Calculate the strength of the sodium hydroxide.*

Answer: The reaction between NaOH and Oxalic acid $[(COOH)_2.2H_2O]$ is:

$$2NaOH + \begin{array}{l} COOH \\ | \\ COOH \end{array} .2H_2O \longrightarrow \begin{array}{l} COONa \\ | \\ COONa \end{array} .2H_2O + 2H_2O$$

We know, $\dfrac{V_{acid} \times S_{acid}}{V_{base} \times S_{base}} = \dfrac{1}{2}$

Here, V_{acid} = 25 mL

S_{acid} = 0.1 M

V_{base} = 20 mL

S_{base} = ?

Putting these values in the above expression, we get,

∴ S_{base} = 0.25 M

Question: *Why do we standardize the supplied NaOH solution first?*

Answer: Because NaOH is a secondary standard substance.

Question: *Write down the name of indicator used in the titration between NaOH and HCl.*

Answer: Methyl Orange.

Question: *Write down the balanced neutralization reaction of NaOH and HCl*

Answer: $HCl\ (aq) + NaOH\ (aq) \rightarrow NaCl\ (aq) + H_2O\ (l)$

Question: *20 ml of a NaOH solution is required for neutralization of 20 ml 0.1(M) HCl acid solution. Calculate the strength of the sodium hydroxide.*

Answer: The reaction between NaOH and HCl is:

$$HCl\ (aq) + NaOH\ (aq) \rightarrow NaCl\ (aq) + H_2O\ (l)$$

From the above equation we can write, $\frac{V_{acid} \times S_{acid}}{V_{base} \times S_{base}} = \frac{1}{1}$

Here, $V_{acid} = 20$ ml $S_{acid} = 0.1$ M

$V_{base} = 20$ ml $S_{base} = ?$

Putting these values in the above expression, we get,

$$\therefore S_{base} = 0.1 \text{ M}$$

Question: *How does a redox indicator works?*

Answer: An oxidation-reduction (redox) indicator is a compound which exhibits different colors in the oxidized and reduced forms:

$$In_{ox} + ne^- \leftrightarrow In_{red}$$

One of the best redox indicators is the ortho-phenanthroline ferrous ion.

Question: *What is the difference between acid base and a redox indicator?*

Answer: An acid-base indicator is either weak acid or weak base and had a different color form that of its ions:

$$HIn \leftrightarrow H^+ + In^-$$
$$InOH \leftrightarrow In^+ + OH^-$$

But, an oxidation-reduction (redox) indicator is a compound which exhibits different colors in the oxidized and reduced forms:

$$In_{ox} + ne^- \leftrightarrow In_{red}$$

Question: *What type of standard is $K_2Cr_2O_7$?*

Answer: It is a primary standard.

Question: *What type of titration you are performing?*

Answer: It is a redox titration.

Question: *What is the function of starch?*

Answer: Starch is used as an indicator to detect the end point of the iodometric titration. Starch reacts with iodine in the presence of iodine to form an intensely blue colored complex, which is visible at very low concentration of iodine.

Question: *What do you mean by iodimetry and iodometry?*

Answer: Iodimetry

Iodimetry covers titrations with a standard solution of iodine, i.e.; the process of titration in which used standard iodine solution. It is called the direct titration method.

Iodometry

Iodometry deals with the titration of iodine liberated in chemical reactions. It is called the indirect titration method.

Question: *Write the basic principle of iodometric titration.*

Answer: The basic principle of iodometric titration is-

1 g equivalent of oxidizing agent = 1 g equivalent of iodine

= 1 g equivalent of reducing agent

Question: *Write the principle error in titrations involving iodine.*

Answer: Two important sources of error in titrations involving iodine are-

1. loss of iodine owing to its appreciable volatility.
2. acid solution of iodine are oxidized by oxygen of the air.

Question: *In order to determine the amount of iron in Mohr's salt what standard solution we use in this experiment?*

Answer: Potassium dichromate ($K_2Cr_2O_7$) solution.

Question: *Write the balanced equation for the estimation of Cu^{2+} ions by iodometric titration.*

Answer:

$2CuSO_4 + 4KI = 2CuI + I_2$

$2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2NaI$

Therefore,

$2\ CuSO_4 \equiv I_2 \equiv 2Na_2S_2O_3$

$1\ \text{mole}\ CuSO_4 \equiv 1\ \text{mole}\ Na_2S_2O_3$

Question: *15 ml of 0.02 (M) $Na_2S_2O_3$ solution react complete with 10 ml of copper solution. Calculate the amount of Cu^{2+} ion 10 ml solution and per liter of solution.*

Answer: Volume of $Na_2S_2O_3$ required to titrate 10 ml of sample = 15 mL

1 mole $CuSO_4 \equiv$ 1 mole $Na_2S_2O_3$

1000 mL 1M $Na_2S_2O_3$ contain = 63.54 g Cu^{2+} ion

15 mL of 0.02 M $Na_2S_2O_3$ = (15×0.02 / 1000) mole Cu^{2+}
= (15×0.02 / 1000) ×63.54 g Cu^{2+}
= 0.019 g Cu^{2+}

10 ml of sample contains = 0.019 g Cu^{2+}

1000 ml of sample contains = (0.019×1000) / 10 g 0.019 g Cu^{2+}
= 1.90 g 0.019 g Cu^{2+} / L

Question: *What type of reaction occurs in estimation of iron experiment?*

Answer: A redox reaction.

Question: *What indicator do we use here?*

Answer: Diphenylamine sulphonate.

Question: *What is the color, which indicates the end point in estimation of iron experiment?*

Answer: The formation of intense purple or violet blue coloration indicates the end point.

Question: *Write down the balanced oxidation-reduction reaction which is used for the calculation of the amount of Fe^{2+} in this experiment.*

Answer: $K_2Cr_2O_7 + 7H_2SO_4 + 6FeSO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$

Ionic form,
$6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \rightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$

Question: *25 ml of 0.02 (M) $K_2Cr_2O_7$ solutions react complete with 10 ml of Mohr's salt solution. Calculate the amount of Fe^{2+} in 10 ml solution and in per liter of solution.*

Answer: Volume of $K_2Cr_2O_7$ required to titrate 10 ml of sample = 25 mL

1mole $K_2Cr_2O_7$ = 6 mole Fe^{2+}

1000 ml of 1M $K_2Cr_2O_7$ = 6 mole Fe^{2+}

25 mL of 0.02 M $K_2Cr_2O_7$ = (25×6×0.02 / 1000) mole Fe^{2+}
= (25×6×0.02 / 1000) ×55.84 g Fe^{2+}
= 0.1675 g Fe^{2+}

10 ml of sample contains = 0.1675 g Fe^{2+}

1000 ml of sample contains = (0.028×1000) / 10 g Fe^{2+}

= 16.75 g Fe^{2+} / L

Question: *Why we don't have to use any indicator in standardization of $KMnO_4$ solution with standard ($Na_2C_2O_4$) solution experiment?*

Answer: Because potassium permanganate ($KMnO_4$) acts as a self indicator.

Question: *Why we have to heat the experiment solution?*

Answer: Because the reaction between potassium permanganate ($KMnO_4$) and sodium oxalate ($Na_2C_2O_4$) in acidic medium is slow.

Question: *What type of titration have you done in standardization of $KMnO_4$ solution with standard ($Na_2C_2O_4$) solution experiment?*

Answer: It is a "redox", i.e.; oxidation-reduction titration.

Question: *What solution we take in burette in standardization of $KMnO_4$ solution with standard ($Na_2C_2O_4$) solution experiment?*

Answer: Potassium permanganate ($KMnO_4$).

Question: *Calculate the molarity of a 250 ml solution, which contains 1.7310 gm of sodium oxalate. The volume of potassium permanganate solution required to titrate 20 ml of this sodium oxalate solution is 21.5 ml. What is the molarity of the permanganate solution?*

Answer: Molecular weight of sodium oxalate ($Na_2C_2O_4$) = 23×2 +12×2 + 16×4 = 134

250mL solution contains 1.7310 gm of sodium oxalate.

1000 mL solution contains = (1000×1.7310)/250

= 6.924 gm of sodium oxalate.

Concentration of sodium oxalate solution = 6.924 / 134

= 0.05

The reaction between $KMnO_4$ and $Na_2C_2O_4$ is:

$$Na_2C_2O_4\ (aq) + H_2SO_4\ (aq) \rightarrow H_2C_2O_4(aq) + Na_2SO_4\ (aq)$$

$$2KMnO_4\ (aq) + 5H_2C_2O_4\ (aq) + 3H_2SO_4\ (aq) \rightarrow 2MnSO_4\ (aq) + K_2SO_4\ (aq) + 10CO_2 + 8H_2O\ (l)$$

From the above equation we can write,

2 mole $KMnO_4$ (aq) ≡ 5 mole $H_2C_2O_4$ (aq) ≡ 5 mole $Na_2C_2O_4$ (aq)

$$5 \times V_1 \times S_1 = 2 \times V_2 \times S_2$$

Here,

Volume of permanganate solution, $V_1 = 21.5$ ml

Strength of permanganate solution, $S_1 =$?

Volume of sodium oxalate solution, $V_2 = 20$ml

Strength of sodium oxalate solution, $S_2 = 0.05$ M

Putting these values in the above expression, we get,

$\therefore S_1 = 0.0186$ M