

Pharmaceutical Analysis . Unit 1

PREPARATION AND STANDARDIZATION OF VARIOUS MOLAR SOLUTIONS.

1. Oxalic acid (COOH)₂

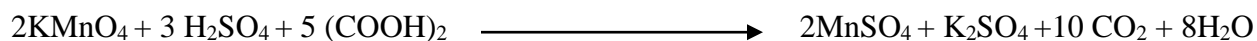
Oxalic acid is available in pure state and its standard solutions can, therefore, be prepared by the direct method.

Eq. wt. of hydrated oxalic acid (C₂H₂O₄.2H₂O), being 63 its 0.1N solution would contain 6.3 gm/litre, and 0.05 N solution would contain 3.15 gm/litre.

These standard solutions are employed to find the strength of solutions of alkalies (NaOH and KOH) whose standard solutions cannot be prepared by the direct method.

PRINCIPLE – REDOX TITRATION

KMnO₄, potassium permanganate is a strong oxidizing agent. Oxalic acid is oxidized by potassium permanganate in acidic solution to produce CO₂ and H₂O



HCl cannot be used in place of sulphuric acid as it readily get oxidized to chlorine in presence of KMnO₄

Preparation Of 0.1N Oxalic Acid Solution

Weigh accurately 6.3 gm of oxalic acid & dissolve in distilled water & finally make up the volume to one liter in a volumetric flask.

Standardization Of 0.1 N Oxalic Acid

- Clean and dry all the glasswares as per standard laboratory procedure.
- Take 20 ml of prepared oxalic acid in a conical flask
- Add 5 ml of sulphuric acid, and warm at 70⁰C
- Rinse the burette with distilled water and pre rinse with the portion of potassium permanganate soln.
- Start the titration with 0.1N KMnO₄ until the end point.
- End point is the appearance of pink colour that persists for more than 30 seconds.
- Record the reading repeat the titration 3 times to get the precise values.

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Calculation

$$\text{Normality of Oxalic Acid} = \frac{\text{Weight Taken} \times \text{Expected Normality}}{\text{Equivalent Weight Factor} \times \text{Titre Value}}$$

Sl no	Content in conical flask	Burette reading		Indicator/color change	Volume used.
		Initial	Final		
1.	20ml oxalic acid + 5ml Sulphuric acid			KMnO ₄ /persistence of pink colour.	
2.					

2. SODIUM HYDROXIDE

Sodium hydroxide is hygroscopic and absorbs water from the air when you place it on the balance for massing. This water will prevent you from being able to find the exact mass of sodium hydroxide.

In order to determine the exact concentration of a sodium hydroxide solution you must standardize it by titrating with a solid acid that is not hygroscopic.

Potassium hydrogen phthalate, KHC₈H₄O₄ (abbreviated KHP/PHP), is a non-hygroscopic, crystalline, solid that behaves as a monoprotic acid.

It is water soluble and available in high purity. Because of its high purity, you can determine the number of moles of KHP directly from its mass and it is referred to as a primary standard. You will use this primary standard to determine the concentration of a sodium hydroxide solution

PRINCIPLE – ACID BASE TITRATION

An acid-base titration is a procedure used to compare the amount (moles) of acid in one sample with the amount (moles) of base in another. In this laboratory exercise you will carry out such a titration to standardize (determine the exact concentration of) a NaOH solution by measuring accurately how many milliliters of it are required to exactly neutralize a known amount of acid. A burette filled with the titrant (NaOH solution) is

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used to measure the volume of NaOH solution added to the known amount of acid in a flask. An indicator is added to signal the endpoint has been reached

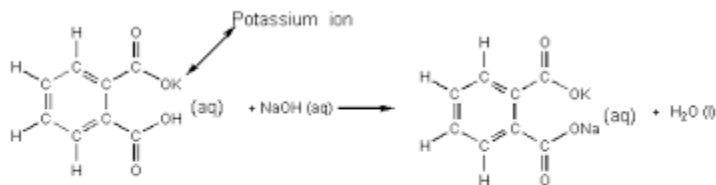


Figure 1. Neutralization reaction of potassium hydrogen phthalate with sodium hydroxide forming sodium potassium phthalate and water.

Procedure

1. Preparation of 0.1 M NaOH

- Weigh about 402 g of sodium hydroxide in a 1000ml volumetric flask. Add water mix the solution well and make up to 1000ml with distilled water

2. Standardization of 0.1M Sodium hydroxide

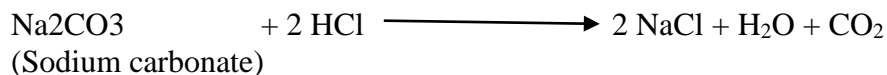
- Weigh accurately about 0.500g of Pottasium hydrogen phthalate, previously powdered and dried at 120 °C for 2 hrs and dissolve in 75ml of CO₂ free water in a conical flask.
- Add 0.1ml phenolphthalein solution
- Fill the burette with prepared 0.1M NaOH
- Titrate the PHP with 0.1M NaOH until thr colour change of a permanent pink colour.
- Repeat the titration for 3 times to get the precise reading
- Equivalent weight factor of PHP for 0.1 M NaOH = 0.020422 g

Calculation

$$\text{Normality of Sdoium hydroxide} = \frac{\text{Weight Taken} \times \text{Expected Normality}}{\text{Equivalent Weight Factor} \times \text{Titre Value}}$$

3. HYDROCHLORIC ACID

Principle- A known concentration or strength of sodium carbonate is titrated directly with HCl. End point can be detected by using methyl orange or methyl red indicator. Reaction involves,



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Preparation of 0.1 M HCl

Pipette out 8.5 ml of HCl and dilute upto 1000 ml with distilled water.

Standardization of 0.1 M HCl.

- Clean and dry all the glasswares as per standard laboratory procedure.
- Rinse the burette with distilled water and again rinse the same with a portion of 0.1M HCl. This is necessary to ensure that all of the solution in the burette is of desired solution not of diluted or contaminated solution.
- Weigh accurately 0.150 g of sodium carbonate heated previously for 1 hr, at 270 °C and transfer it into a conical flask.
- Add 20 ml of distilled water and shake well or sonicate for 5 min.
- Add 2 drops of methyl orange indicator
- Titrate with 0.1 M HCl by adding small quantities of HCl soln from burette.
- End point is the appearance of pink colour that persists for 30 seconds.
- Repeat the titration for 3 times to get the precise reading
- Equivalent weight factor of sodium carbonate for 0.1 M HCl= 0.00529 g

Calculation

$$\text{Normality of 0.1 N HCl} = \frac{\text{Weight Taken} \times \text{Expected Normality}}{\text{Equivalent Weight Factor} \times \text{Titre Value}}$$

4, SODIUM THIOSULPHATE

Sodium thiosulfate is an inorganic sodium salt composed of sodium and thiosulfate ions in a 2:1 ratio. It has a role as an antidote to cyanide poisoning, a nephroprotective agent and an antifungal drug.

- Synonyms: SODIUM THIOSULFATE
- Molecular Formula: $\text{Na}_2\text{O}_3\text{S}_2$ or $\text{Na}_2\text{S}_2\text{O}_3$

It is typically found in its pentahydrate form which is either white in colour, or colourless altogether. This pentahydrate of sodium thiosulfate is described by the following chemical formula: $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$.

In its solid form, it is a crystalline solid which has a tendency to readily lose water. Sodium thiosulfate is readily soluble in water and is also referred to as sodium hyposulfite.

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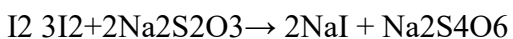
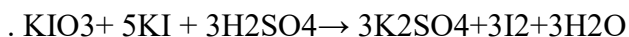
PRINCIPLE

-Redox Titration (IODOMETRY)

The principle of standardization of sodium thiosulphate is based on redox iodometric titration with potassium iodate (primary standard).

Potassium iodate is a strong oxidizing agent, it is treated with excess potassium iodide in acidic media which liberates iodine which is back titrated with sodium thiosulphate.

Uniformity of reactions between iodine and sodium thiosulphate forms basis for utilizing the standard solution of iodine in the analysis of sodium thiosulphate.



PROCEDURE:

Preparation of 0.1N Sodium thiosulphate

- Dissolve 24.8g of sodium thiosulphate pentahydrate($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in 800 ml of freshly boiled and cooled water and mix thoroughly by shaking for approximately 15 minutes.
- Make up the volume to 1000 ml.

Preparation of 0.1N Potassium Iodate

- Weigh accurately about 356 mg of KIO_3 and dissolve in 100 ml distilled water.

Preparation of Starch indicator

- Take 1 gm of soluble starch and triturate with 5 ml of water and add it to 100 ml of Boiling water containing 10 mg of Mercuric iodide with continuous stirring.

Standardization of 0.1N sodium thiosulphate

- Take 10 ml of Potassium Iodate solution .
- Add 2 gm of Potassium Iodide and 5 ml of dilute H_2SO_4 , keep it in dark for 10 minutes.
- add 2 to 3 drops of starch indicator and titrate with sodium thiosulphate using starch solution as indicator until the blue colour is disappeared.
- Record the reading , Repeat the titration for 3 times to get the precise reading
- Equivalent weight factor of sodium carbonate for 0.1 M HCl = 0.00529 g

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Calculation

$$\text{Normality of 0.1 N sodium thio sulphate} = \frac{\text{Weight Taken} \times \text{Expected Normality}}{\text{Equivalent Weight Factor} \times \text{Titre Value}}$$

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