Experiment

Preparation and standardization of Sodium thiosulfate

Aim:- To Prepare and standardization ~0.1 M sodium thiosulfate solution.

Reference:-Indian Pharmacopoeia by Ministry of Health and Family Welfare, Gov. of India, Volume I 2007, P-316.
Practical book of Pharmaceutical Chemistry, Fourth edition edited by A.H Beckett, J.B Stenlake, CBS Publishers and Distributors, 2005 P-187.

Requirements:

- **Chemical Requirement:-** Sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$ Molar mass ≈ **248.18 g·mol⁻¹**, Sodium carbonate (Na_2CO_3), Potassium iodate (KIO_3) Molar mass ≈ **214.00 g·mol⁻¹**, primary standard (dry at 120 °C for 1–2 h; cool in desiccator), Potassium iodide (KI), Hydrochloric acid ~1 M (or sulfuric acid ~1 M), Starch indicator, fresh 0.5% w/v, distilled water (CO_2-free).
- **Glassware Requirement:-** Volumetric flask (1 L), analytical balance (±0.1 mg), 50 mL burette, pipettes (10, 25 mL), conical flasks (250–500 mL), measuring cylinder, amber storage bottle, glass rod, funnel.

Principle:

1. In acidified iodide, iodate liberates iodine:
   $\mathbf{IO_3^- + 5\ I^- + 6\ H^+ \rightarrow 3\ I_2 + 3\ H_2O}$
2. Iodine is titrated with thiosulfate:
   $\mathbf{I_2 + 2\ S_2O_3^{2-} \rightarrow 2\ I^- + S_4O_6^{2-}}$

Stoichiometric link: 1 mol $KIO_3 \equiv 6$ mol $S_2O_3^{2-}$.

Hence, from a weighed KIO_3, you can compute the exact molarity/normality of thiosulfate

Preparation of ~0.1 M $Na_2S_2O_3$

1. **Weigh** 24.8 g $Na_2S_2O_3 \cdot 5H_2O$ into a beaker.
2. **Add** 0.10 g Na_2CO_3.
3. **Dissolve** in ~500 mL freshly boiled & cooled water.
4. **Transfer** to a 1 L volumetric flask; rinseings included.
5. **Make up** to the mark with the same water; **mix well**.
6. **Store** in an amber bottle, well-stoppered, **24 h** before use.

Approximate molarity calculation:

$$M = \frac{\text{mass (g)}}{\text{M.W. (g/mol)} \times V\text{(L)}} = \frac{24.8}{248.18 \times 1.000} = 0.0999\ M \approx 0.1\ M$$

Standardization with KIO_3 (Primary Standard)

1. Accurately weigh about **0.037 g KIO_3** into a conical flask.
2. Add ~2 g KI and 25–30 mL distilled water.
3. Add 10 mL 1 M HCl → iodine (brown solution) liberated.
4. Titrate with $Na_2S_2O_3$ solution until pale yellow.
5. Add 1–2 mL starch → deep blue color appears.
6. Continue titration until sharp colorless endpoint.
7. Repeat for 3 concordant readings (≤0.1 mL difference)

Observations (example table)

Trial	Mass of KIO_3 (g)	Burette Reading (mL)	Titre (mL)
1	0.03720	0.00 – 15.18	15.18
2	0.03720	0.00 – 15.20	15.20
3	0.03720	0.00 – 15.22	15.22
Mean	0.03720	—	**15.20**

Calculations

Step 1. Moles of KIO_3 used

$$n(KIO_3) = \frac{m}{M.W.} = \frac{0.03720}{214.00} = 1.738 \times 10^{-4}\ mol$$

Step 2. Moles of $Na_2S_2O_3$ required

(1 mol KIO_3 → 6 mol $Na_2S_2O_3$)

$$n(S_2O_3^{2-}) = 6 \times n(KIO_3) = 6 \times 1.738 \times 10^{-4} = 1.043 \times 10^{-3}\ mol$$

Step 3. Volume of thiosulfate used

$$V = 15.20\ mL = 0.01520\ L$$

Step 4. Molarity of $Na_2S_2O_3$

$$M = \frac{n}{V} = \frac{1.043 \times 10^{-3}}{0.01520} = 0.0686\ M$$

Result:- The Molarity of given $Na_2S_2O_3$ solution is …… M.

Viva Voce Questions

1. Why is starch used as an indicator?
2. What is the reaction involved in the standardization process?
3. How do you calculate the exact molarity of sodium thiosulfate solution?
4. Why is it important to use freshly boiled and cooled water for preparation?
5. How is the endpoint of the titration detected?