Experiment

Preparation and standardization of Sulphuric acid

Aim:- To Prepare and standardization of 0.5M Sulphuric acid solution.

Reference:-Indian Pharmacopoeia by Ministry of Health and Family Welfare, Gov. of India, Volume I 2007, P-313-316.

Practical book of Pharmaceutical Chemistry, Fourth edition edited by A.H Beckett, J.B Stenlake, CBS Publishers and Distributors, 2005 P-140.

Requirements:

- Chemical Requirement:- Concentrated sulfuric acid, Sodium carbonate (Na₂CO₃), Indicator: methyl orange (2–3 drops), Distilled water.
- Glassware Requirement:- Volumetric flask 1 L, beakers, 25–50 mL burette, pipettes, conical flasks (250–500 mL), analytical balance (±0.1 mg), graduated cylinder, glass rod, funnel, ice bath, fume hood.

Principle:

Concentrated H₂SO₄ is diluted to the required molarity and then its exact concentration is determined by titration with a primary standard (Na₂CO₃).

Reaction:

$$Na_2CO_3 + H_2SO_4 \rightarrow Na_2SO_4 + H_2O + CO_2 \uparrow$$

Procedure

A. Preparation of approximate 0.5 M H₂SO₄

- 1. Place ~500 mL distilled water in a 1 L beaker in an ice bath (to control heat).
- 2. Take **27.2 mL** concentrated H₂SO₄.
- 3. With stirring, **slowly add the acid to the water** dropwise at first. Allow temperature to fall between additions. Do not add water to acid.
- 4. After all acid is added and solution cooled to near room temp.
- 5. Make up to the 1 L mark with distilled water
- 6. Cool to room temperature; label: "~0.5 M H₂SO₄ to be standardized", date and preparer.

B. Drying primary standard (Na₂CO₃)

1. If required, dry Na₂CO₃ at 110 °C for 1–2 h, cool in desiccator. (Record mass after drying.)

C. Standardization (titration)

- 1. Accurately weigh ~0.50-0.60 g of dried Na₂CO₃.
- 2. Dissolve weighed Na₂CO₃ in ~250 mL distilled water in a 500 mL conical flask.
- 3. Add 2–3 drops methyl orange indicator.
- 4. Rinse and fill burette with prepared H₂SO₄ solution; note initial burette reading.
- 5. Titrate with H₂SO₄, swirling constantly, until colour changes from yellow → orange/red (methyl orange endpoint). Record final burette reading; compute titre V (mL). Perform three concordant titrations (within 0.10–0.20 mL). Use mean V.

Observations / Data table (example layout)

Trial	Mass Na₂CO₃ (g)	Initial burette (mL)	Final burette (mL)	Titre V (mL)
1	0.5500	0.00	32.10	32.10
2	0.5500	0.00	32.25	32.25
3	0.5500	0.00	32.05	32.05
Mean	_	-	9	32.13

Calculations (example using above data)

- $$\begin{split} &\text{Molar mass Na}_2\text{CO}_3 = 105.99 \text{ g·mol}^{\text{-1}}. \\ &\text{Moles Na}_2\text{CO}_3 = \frac{0.5500}{105.99} = 0.005188 \text{ mol}. \end{split}$$
- From stoichiometry, moles $H_2SO_4 = 0.005188$ mol.
- Volume of H_2SO_4 used = mean titre V=32.13 mL = 0.03213 L.
- Molarity $M=rac{0.005188}{0.03213}=0.1615~ ext{M}.$

Result:- The molarity of prepared H2SO4 solution is M.

Viva-Voce Questions

- 1. Why do we prepare approximate H₂SO₄ solution and then standardize it?
- 2. Why do we use methyl orange indicator instead of phenolphthalein?
- 3. How will you calculate the molarity of H₂SO₄ from titration data?
- 4. What is the difference between molarity and normality?
- 5. Why should acid be added to water, and not water to acid?