EXPERIMENT 2 PREPARATION OF STANDARD VOLUMETRIC SOLUTIONS

Structure

2.0 OBJECTIVES

After attending to this experiment, we shall be able to:

- prepare standard solutions.

2.1 INTRODUCTION

A solution of known normality is called a standard solution. Its concentration is determined by a process known as standardization. If you have a primary standard (a compound which is very pure, stable, non-hygroscopic, and with a high molecular weight), you can prepare a standard solution simply by dissolving a known amount of the compound in a known volume of liquid. If you don't have such a compound, you'll have to standardize your solution against a primary standard.

2.2 PREPARATION OF STANDARD VOLUMETRIC SOLUTIONS

2.2.1 Standard Thiocyanate Solution (0.1 N)

Reagents

Ferric alum indicator solution – Saturated solution of $FeNH_4(SO_4).12H_2O$ in water.

Silver nitrate- purified by treating 50 g of $AgNO_3$ in 20 ml boiling water with 5 drops of HNO_3. Heat to dissolve and filter.

Procedure

Dissolve about 7.612 g of ammonium thiocyanate (NH_4SCN) or 9.718 g of potassium thiocyanate (KSCN) in water and dilute to 1000 ml. Standardize the solution as follows:

Pipette 25 ml of standard 0.1 N silver nitrate solution into a 250 ml conical flask, add 5 ml of nitric acid (1:1) and 2 ml of ferric alum indicator solution. Titrate with ammonium thiocyanate or potassium thiocyanate solution from a burette. At first, a white precipitate is produced imparting a milky appearance to the solution, and as each drop of thiocyanate fills in, it produces a pink colour, which quickly disappears on shaking. As the end point approaches, the precipitate becomes flocculent and settles easily. The end point is reached when one drop of the thiocyanate solution produces a reddish brown colour, which remains after shaking vigorously for 1 min. Repeat the titration with two other 25 ml portions of silver nitrate solution. Individual titration should agree within 0.1 ml. Determine the reagent blank. Calculate the normality of the thiocyanate solution on the basis of normality of standard silver nitrate solution.

Calculation

$$\text{Normality of thiocyanate} = \frac{\text{Normality of } AgNO_3 \times 25}{\text{Titre Volume (ml)}}$$

Note : If purified $AgNO_3$ is taken directly for titration, then accurately weigh 0.7 g AgNO3 to 250 ml Erlenmeyer flask and add 75 ml of distilled water, 5 ml of HNO_3 (1:1) and 2 ml ferric alum solution. Titration proceeds as above.

$$\text{Normality of thiocyanate} = \frac{\text{Wt of } AgNO_3 \times 1000 \times \%\text{Purity}}{\text{Titre Volume (ml)} \times 169.87 \times 100}$$

2.2.2 Standard Hydrochloric Acid Solution (0.5 N)

Reagents

Methyl orange - 0.1% in H_2O.

Anhydrous sodium carbonate - Primary standard (NIST certified).

Procedure

Transfer by means of graduated cylinder or a burette 44.5 ml (sp gr 1.18) or 49.1 ml (sp gr 1.16) of concentrated hydrochloric acid (HCl) into a 1000 ml volumetric flask containing about 500 ml of water. Cool and dilute to 1000 ml. Standardize the solution as follows:

Take 0.3 g of anhydrous sodium carbonate into 250 ml conical flask, dilute to 40 ml with water, and add 3 drops of methyl orange. Titrate the sodium carbonate solution against the hydrochloric acid solution to be standardized, continue the addition until the colour due to methyl orange becomes a very faint yellow. Boil the solution and cool again. Continue the titration by adding the acid drop-wise and carefully until the colour of the methyl orange becomes orange or faint pink. The titration is repeated with two other portions of sodium carbonate solution. Individual titration should agree within 0.1 ml. Calculate the normality of the hydrochloric acid as follows.

Calculation

$$\text{Normality of HCl} = \frac{\text{Wt of } Na_2CO_3 \times 1000 \times \%\text{Purity}}{\text{Titre Volume (ml)} \times 53.0 \times 100}$$

2.2.3 Standard Iodine Solution (0.1 M)

Reagents

Arsenious oxide - NIST certified (Dry at 105°C for 1 h immediately before use).

Resublimated iodine - AR grade

Arsenious oxide solution (0.025M) : Accurately weigh 4.95 g of As_2O_3. Dissolve in 50 ml of 1M NaOH in a flask by heating on steam bath. Add same amount of 0.5M H_2SO_4. Cool, quantitatively transfer mixture to 1 litre volumetric flask and dilute to volume. Solution must be neutral to litmus.

Sodium bicarbonate-AR grade.

Starch solution - 0.2% solution in water.

Procedure

Transfer 12.7 g of resublimed iodine to a 250 ml beaker containing 20 g of iodate free potassium iodide and 50 ml of water. Stir until all iodine is dissolved and dilute with water to 1000 ml. Preserve in an amber-colour glass stoppered bottle in a cool place. Standardize the solution as follows:

Transfer accurately 50 ml of As_2O_3 solution to Erlenmeyer flask. Acidify slightly with H_2SO_4 (1:10), neutralize with solid $NaHCO_3$ and add 2 g excess. Titrate with I_2 solution using 2.5 ml starch solution as indicator. Saturate solution with CO_2 at the end of titration by adding 1 ml of H_2SO_4 (1:10) just before end point is reached.

Calculation

$$\text{Molarity of } I_2 \text{ solution} = \frac{\text{Molarity of } As_2O_3 \times 50}{\text{Titre Volume (ml)}}$$

Note: The strength of iodine solution deteriorates on keeping. It shall always be standardized before use.

2.2.4 Standard Potassium Permanganate Solution (0.05 N)

Reagents

Disodium oxalate - NIST certified (105°C for 1 h).

Dilute sulphuric acid - (10% in water).

Potassium permanganate - AR grade.

Procedure

Carefully dissolve 3.2 g of potassium permanganate ($KMnO_4$) in water and dilute to 1000 ml. Boil solution for 1 h. Allow to stand for 2-3 days in an amber-coloured glass stoppered bottle. Filter off any manganese hydroxide through glass wool. Store in amber coloured glass stoppered bottle. Standardize the solution as follows:

Weigh 0.3 g of dried sodium oxalate ($Na_2C_2O_4$) into 250 ml conical flask and dissolve in 40 ml water by slow stirring. Add 50 ml of 10% H_2SO_4 solution to it. Add 20 ml of $KMnO_4$ solution from a burette while shaking till the solution is decolourized and then heat to 55-60°C. At this temperature, complete the titration, by adding $KMnO_4$ solution until faint pink colour persists for 30 sec. Repeat the

titration two more times. Run a blank determination using 50 ml of 10 per cent sulphuric acid. Calculate the normality of potassium permanganate based on the quantity of standard sodium oxalate taken.

Calculation

$$\text{Normality of } KMnO_4 = \frac{\text{Amount of } Na_2C_2O_4\,(g) \times 1000 \times \%\text{Purity}}{\text{Titre Volume (ml)} \times 133.999 \times 100}$$

2.2.5 Standard Silver Nitrate Solution (0.1 N)

Reagents

Sodium chloride - NIST certified (Dried at 110°C for 2h).

Potassium chromate solution (5% solution in water).

Silver nitrate - AR grade.

Procedure

Dissolve 16.9873 g of silver nitrate ($AgNO_3$) in water and dilute to 1000 ml. All apparatus should be thoroughly washed with absolutely chloride free distilled water before use. Store in amber coloured glass-stoppered bottle. Standardize the solution as follows:

Accurately weigh 0.3 g dried sodium chloride (NaCl) into a 250 ml conical flask resting on a white porcelain tile and add 40 ml of water. Add 1 ml of potassium chromate indicator solution and titrate against silver nitrate solution until first precipitable pale-red brown persists after vigorous shaking. Determine the indicator blank correction by adding 1 ml of the indicator to a volume of water equal to the final volume in the titration and then silver nitrate solution until the colour of the blank matches that of the solution titrated. The indicator blank correction should not amount to more than 0.03 to 0.1 ml of silver nitrate. Repeat the titration two more times. Calculate the normality of silver nitrate solution on the basis of quantity of Na_2CO_3 taken for titration.

Calculation

$$\text{Normality of } AgNO_3 = \frac{\text{Amount of NaCl (g)} \times 1000 \times \%\text{Purity}}{\text{Titre Volume (ml)} \times 58.45 \times 100}$$

2.2.6 Standard Sodium Hydroxide Solution (0.1 N)

Reagents

Carbon dioxide free water - Boil water for 20 min and cool with soda lime protection.

Sodium hydroxide - AR grade

Potassium hydrogen phthalate - NIST certified (Dried at 120°C for 2h). Cool in desiccator containing H_2SO_4.

Procedure

Dissolve 4 g of sodium hydroxide (NaOH) in l00 ml of boiled and carbon-dioxide free water in an Erlenmeyer flask and dilute to 1000 ml with water. Store in a polyethylene bottle. Standardize this solution as follows:

Accurately weigh 0.3 g enough dried potassium hydrogen phthalate ($KHC_8H_4O_4$) into 250 ml Erlenmeyer flask and add 50 ml cool CO_2 free H_2O. Titrate to pH 8.6 against NaOH solution using phenolphthalein as indicator or by pH meter. Repeat the titration two more times. Calculate the normality of sodium hydroxide solution on the basis of standard potassium hydrogen phthalate.

Calculation

$$\text{Normality of NaOH} = \frac{\text{g of } KHC_8H_4O_4 \times 1000 \times \%\text{Purity}}{\text{Titre Volume (ml)} \times 204.229 \times 100}$$

2.2.7 Standard Sodium Thiosulphate Solution (0.1 N)

Reagents

Potassium dichromate - NIST certified (Dried at 100°C for 2h)

Potassium iodide - AR grade

Conc. HCl

Sodium thiosulphate - A grade

Starch solution (0.5%): Mix 0.5 g of soluble starch with about 15 ml of water and pour into 100 ml of hot water. Boil for 1-2 min.

Procedure

Weigh 25 g of sodium thiosulphate crystals ($Na_2S_2O_3.5H_2O$), dissolve in water and dilute 1000 ml. Standardize the solution as follows:

Weigh accurately 0.125 g of ground potassium dichromate into a 250 ml conical flask and 25 ml of distilled water. Add 2 g of potassium iodide and 8 ml of concentrated hydrochloric acid. Mix thoroughly and titrate with sodium thiosulphate, swirling the liquid constantly until the brown colour changes to yellowish green. Add 2 ml of starch solution and continue the titration until the colour changes sharply from blue to light green. Repeat the titration two more times. Calculate the normality of sodium thiosulphate solution on the basis of normality of potassium dichromate.

Calculation

$$\text{Normality of } Na_2S_2O_3 \text{ solution} = \frac{\text{g of } K_2Cr_2O_7 \times 1000 \times \%\text{Purity}}{\text{Titre Volume (ml)} \times 49.03 \times 100}$$

2.2.8 Standard Sulphuric Acid Solution (0.1 N)

Reagents

Standard NaOH solution (0.1N)

Phenolphthalein indicator solution: Dissolve 1 g in 100 ml ethanol.

Procedure

Transfer 3 ml of sulphuric acid (sp gr l.84) into a 1000 ml volumetric flask containing about 500 ml of water. When cold, dilute to 1000 ml. Standardize the solution as follows:

Transfer 25 ml of standard 0.1N sodium hydroxide solution into 250 ml conical flask. Titrate with sulphuric acid solution using phenolphthalein indicator. Repeat the titration with two more 25 ml portions of sodium hydroxide solution. The various titration should agree within 0.1 ml. Calculate the normality of sulphuric acid solution on the basis of standard sodium hydroxide solution.

Calculation

$$\text{Normality of } H_2SO_4 = \frac{\text{Normality of NaOH} \times 25}{\text{Titre Volume (ml)}}$$

2.3 PRECAUTIONS

- Use amber-coloured bottle for the storage of standard solutions.
- Calibrated analytical balance and calibrated volumetric glassware should be used.
- Observe standard volumetric solution for any precipitation at the time of use.
- Pure analytical reagent grade chemicals should be used.
- The standard volumetric solutions should not be stored for a longer period of time.
- The strength of the solutions should be checked before use.