Pharmaceutical analysis- BP102T

UNIT: 1 **(Pharmaceutical analysis, its definition and scope, methods expressing techniques)**

CLASS:4

TOPIC: PREPARATIONS AND STANDARDISATION OF VARIOUS SOLUTIONS

Preparation of Concentrated acids

Prepare approximately 0.1 N solutions on the basis of strength diluting it 120 times with distilled water. Then standardize it against standard N/10 Na_2CO_3 using methyl orange as an indicator.

Standardization of Concentrated acids (HCl)

Prepare approximetly 0.1N solution on the basis of the strength given on the label by diluting it 120 times with distilled water. Then standardize it against standard N/10 NaOH which is already

Standardized against N/10 oxalic acid using phenolphthalein indicators.

Preparation of sulphuric acid (H_2SO_4)

Concentrated H2So4 is very corrosive in nature; therefore, it should be handled carefully. And always remember add acid to water under cold condition this is done to avoid pumping due to the heat generated.

Standardization of Sulphuric Acid H_2SO_4

For the preparation of N/10 H_2SO_4, take 10 ml of concentrated H_2SO_4 (usually about 36 N), dilute 36 times by adding acid in small quantity to distilled water in a cold water bath, to make it 1N and then dilute this 1N solution further 10 times to make it N/10. Then standardize against standard N/10 NaOH or N/10 KOH using phenolphthalein indicator.

Preparation of nitric acid (HNO3)

Take 10 ml of concentrated nitric acid HNO3 (about 16N), and dilute 16 times by adding acid to distilled water to make it 1N and then dilute this 1N solution futher 10 tiimes to make it N/10 then standerize agaianst standard N/10 KOH using phenolphthalein indicator.

Preparation and standardization of EDTA solutions

1. Preparation of 0.01 M EDTA solution: Dissolve 3.8 g of disodium ethylene diamine dihydrogen tetraacetate (EDTA, M.Wt. 372.25) in distilled water and volume is made to 1 litre. Mix it well, store in polyethylene reagent bottle. It is standardized against 0.01 M $CaCO_3$ or $CaCl_2$.
2. Preparation of 0.01 M $CaCl_2$ solution: Prepare standard Ca solution (1 ml = 1 mg $CaCO_3$, M.wt. 100) by weighing 1 g $CaCO_3$ into 500 ml conical flask or beaker and adding dilute HCl through funnel until $CaCO_3$ is dissolved. Add 20 ml water, boil to expel CO_2 and cool. Add few drops of methyl red indicator and adjust colour intermediate orange (brownish red) with dilute NH_4OH or HCl as required. Transfer quantitatively to 1 L volumetric flask and make up volume to the mark. Shake it well and store it well and store in air-tight reagent bottle.
3. Erichrome Black T indicator: Dissolve 0.5 g of Erichrome black T in 100 ml of triethanolamine. Or 0.4 g in 100 ml methanol.
4. Buffer solution: Dissolve 16.9 g NH_4Cl in 143 ml NH_4OH, and dilute to 250 ml with water. Store in tightly stoppered Pyrex of plastic bottle. Dispense from bulb-operated pipette. Discard after 1 month or when 1-2 ml added to sample fails to produce pH 10.0±0.1 at end point titration.

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Preparation of 0.1N sodium thiosulphate solution ($Na_2SO_3.5H_2O$)

Dissolve approximately 24.8 gm of sodium thiosulphate crystals in previously boiled and cooled distilled water and make the volume to 1000 ml. Store the solution in a cool place in a dark colored bottle. After storing the solution for about two weeks, filter if necessary and standardize as follows.Weigh accurately about 5.0 gm of finely ground potassium dichromate which has been previously dried to a constant weight at 105 ± 2° in to a clean 1.0 litre volumetric flask. Dissolve in water make up to the mark; shake thoroughly and keep the solution in dark place. Pipette 25.0 ml of this solution into a clean glass stoppered 250 ml conical flask. Add 5.0 ml of concentrated hydrochloric acid and 15.0 ml of 10% potassium iodide solution. Allow to stand in dark for 5 minutes and titrate the mixture with the solution of sodium thiosulphate using starch solution as an indicator towards the end. The end point is taken when blue color changes to green. Calculate the normality (N) of the sodium thiosulphate as follows:

$$N = \frac{25W}{49.03\ V}$$

W= Weight in g of the potassium dichromate

V= Volume in ml of solution thiosulphate solution required for the titration

Preparation of 0.1N ceric ammonium sulphate $(NH_4)_4Ce(SO_4)_4 2 H_2O$

66gm of ceric ammonium sulphate was dissolved with gentle heat in a mixture of 30 ml of sulphuric acid and 500 ml of water. The mixture was cooled and filtered. The resulting solution was diluted to 1000ml with water

Standardisation of 0.1 N Ceric Ammonium Sulphate

1. About 0.2 gm of Arsenic trioxide which was previously dried for about an hour was accurately weighed and transferred into a 500 ml conical flask.
2. The inner walls of the flask were washed with 100 ml of water and mixed thoroughly
3. Then 300 ml of dil. sulphuric acid , 0.15 ml of osmic acid, 0.1 ml of ferroinsulphate indicator were added
4. Titration was carried out until pink colour of solution changed to pale blue or yellowish green colour
5. Each ml of 0.1 N ceric ammonium sulphate ~ 0.6326 gm of ceric ammonium sulphate ~ 4.946 grams of arsenic trioxide