Pharmaceutical analysis- BP102T

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

CLASS:5

TOPIC: Preparation and standardization of various sample solutions

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

Standardization 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 ferroin sulphateindicator 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

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 madeto 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 welland 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.

5. **Standardization of EDTA solution**

6. Rinse and then fill burette with prepared EDTA solution. Pipette 25 ml of standard $CaCO_3$ solution into 250 ml Erlenmeyer flask, add 1 ml ammonia buffer (to raise the pH as reaction takes place at high pH) and 3-4 drops of Erichrome black T indicator. Titrate the EDTA solution until colour changes from wine red to dark blue with no reddish tingeremaining. Calculate the molarity of EDTA ($M_1V_1 = M_2V_2$), if excess follows the procedure for the standardization, recheck the molarity and it should be 0.01 M.

Preparation of Potassium Permanganate

Potassium Permanganate 0.1 N: Dissolve 3.3 g of reagent grade potassium permanganate (KmnO4) in 1 L of purified water and heat on a steam bath for two hrs. Cover and allow to sample

stand for 24 hrs. Filter through a fine porosity sintered glass crucible, discarding the first 25 mL. Store in a glass-stoppered, amber-colored bottle. Avoid exposure to direct sunlight; cover the neck of the bottle with a small beaker as a protection against dust. If manganese dioxide precipitates on standing, refilter and re standardize before use

Standardization of Potassium Permanganate

Potassium Permanganate 0.1 N: Weigh accurately 0.2-0.3 g sodium oxalate (Na2 C2 O4) (dried 2 hrs., 105-110 °C) National Institutes of Science and Technology, U. S. Department of Commerce. Cool in a desiccator and transfer quantitatively to a 600 mL beaker. Add 250 mL of purified water (freshly boiled and cooled) and 10 mL sulfuric acid (96% H2 SO4 , sp g1.84). Add rapidly from a buret about 95% of the theoretical quantity of potassium permanganate solution needed; stir until the solution is clear. Heat the solution to 55-60 °C (Maintain temperature range during titration.) and complete the titration by slow dropwise addition until the appearance of a pink color which persists for 30 secs. Determine and subtract a blank titration run at 55-60 °C on a mixture of 250 mL of purified water (freshly boiled and cooled) and 10 mL of concentrated sulfuric acid.

Very Short Answer Questions:

1. How to Prepare 0.1N of Potassium permanganate solution.
2.write a short note on Ceric ammonium Sulphate Preparation.

Short Answer Questions:

1.Explain the principle and procedure Potassium permanganate