

I SEMESTER (1st year B.Pharm)

PHARMACEUTICAL ANALYSIS

PRACTICAL LAB MANUAL

LIST of EXPERIMENTS IN PHARMACEUTICAL ANALYSIS LAB

SL. NO.	EXPERIMENT NAME	Page No.
1	PREPARATION AND STANDARDIZATION of 0.1(N) SODIUM HYDROXIDE SOLUTION with 0.1(N) OXALIC ACID SOLUTION	2
2	PREPARATION AND STANDARDIZATION of 0.1(N) SULPHURIC ACID SOLUTION with 0.1(N) SODIUM HYDROXIDE SOLUTION	3
3	PREPARATION AND STANDARDIZATION of 0.1(M) SODIUM THIOSULPHATE SOLUTION by USING 0.1(M) POTASSIUM DICHROMATE SOLUTION	4
4	ASSAY of SODIUM CHLORIDE by USING 0.1(M) AMMONIUM THIOCYANATE SOLUTION	5
5	ASSAY of SODIUM BENZOATE with 0.1(N) PERCHLORIC ACID	6
6	LIMIT TEST of CHLORIDE ION in A SUPPLIED SAMPLE of D-GLUCOSE	7
7	LIMIT TEST of SULPHATE ION in A SUPPLIED SAMPLE of BORIC ACID	8
8	LIMIT TEST of IRON in A SUPPLIED SAMLE of SODIUM BI CARBONATE	9

EXPERIMENT NO- 1.

PREPARATION AND STANDARDIZATION of 0.1 (N) SODIUM HYDROXIDE SOLUTIONS with 0.1 (N) OXALIC ACID SOLUTIONS

REQUIREMENTS-

Funnel, Pipette, Beaker, Volumetric flask, Burette, Oxalic acid, Sodium Hydroxide, Phenolphthalein indicator solution, Distilled water

Procedure-

I. Preparation of 0.1 N Sodium Hydroxide solutions - 0.4 gm of Sodium Hydroxide was weighed & dissolved in few ml of distilled water. Then the volume was making up to 100 ml.

II. Preparation of 0.1 N Oxalic acid solutions - 0.63 gm of Oxalic acid was weighed out & dissolves in few ml of distilled water. Then the volume was making up to 100 ml.

III. Standardization of 0.1 N Sodium Hydroxide solutions- Pipette out 20 ml of Oxalic acid solution from volumetric flask to the conical flask. Then fill the burette with 0.1N NaOH solution up to 50 ml. Add 2 drops of Phenolphthalein indicator solution to the oxalic acid solution (in conical flask). After that continue the titration and end point is colourless to pink colour solution.

EXPERIMENT NO- 2.

PREPARATION AND STANDARDIZATION of 0.1(N) SULPHURIC ACID SOLUTIONS with 0.1(N) SODIUM HYDROXIDE SOLUTIONS

REQUIREMENTS-

Funnel, Pipette, Beaker, Volumetric flask, Burette, Sulphuric acid, Sodium Hydroxide, Methyl orange indicator solution, Distilled water

Procedure-

I. Preparation of 0.1 N Sodium Hydroxide solutions - 0.4 gm of Sodium Hydroxide was weighed & dissolved in few ml of distilled water. Then the volume was making up to 100 ml.

II. Preparation of 0.1 N Sulphuric acid solutions – 0.3 ml of Sulphuric acid dissolved in few ml of distilled water .Then the volume make up to 100 ml with distilled water.

III. Standardization – Pipette out 20 ml of NaOH solution from volumetric flask & placed in a conical flask, add 2-3 drops of methyl orange solution to the conical flask.

Then fill the burette with 0.1 N Sulphuric acids up to 50 ml.

Titration was continuing. End point is detected by the colour change of solution that is yellow to red.

EXPERIMENT NO- 3.

PREPARATION AND STANDARDIZATION of 0.1(M) SODIUM THIOSULPHATE SOLUTIONS by USING 0.1(M) POTASSIUM DICHROMATE SOLUTIONS

REQUIREMENTS-

Funnel, Pipette, Beaker, Volumetric flask, Burette, Sodium thiosulphate solutions, Potassium dichromate solutions, Starch solutions, Distilled water

Procedure-

I. Preparation of 0.1 M Sodium thiosulphate Solution- weighing about 2.5 gm of sodium thiosulphate and is dissolved in 20 ml of distilled water in the volumetric flask. Add 0.02 gm of Sodium carbonate to the volumetric flask. Then volume make up to 100 ml with distilled water. Keep the solutions rest for 1 hour.

II. Standardization of Sodium thiosulphate Solution- 210 mg of Potassium di chromate was weighed and dissolved in 100 ml of distilled water in a 500 ml glass stopper conical flask. Dissolve the solid, remove stopper & quickly add 5 gm of Potassium iodide, 2 gm of Sodium bicarbonate & 5 ml Hydrochloric acid.

Insert the stopper gently in the flask, shake the mixture & allow standing in the dark for exactly 10 minutes. Then 20 ml of this solution was pipetting out & taken into a 50 ml of conical flask & titrated with 0.1 M Sodium thiosulphate solution until the solution is yellowish in colour. Add 3 drops of starch indicator & continue the titration until the blue colour is appeared.

EXPERIMENT NO- 4.

ASSAY of SODIUM CHLORIDE by USING 0.1(M) AMMONIUM THIOCYANATE SOLUTIONS

REQUIREMENTS-

Funnel, Pipette, Beaker, Volumetric flask, Burette, Sodium Chloride, Ammonium thiocyanate solutions, Silver nitrate solutions, Ferric ammonium sulphate solutions, Nitric acid, Di-butyl Phthalate, Distilled water

Procedure –

I. Preparation & standardization of 0.1M Ammonium thiocyanate solutions -

Weighing about 0.76 gm of Ammonium thiocyanate & dissolved it in few ml of distilled water. Final volume is making up to 100 ml with distilled water. Pipetting about 30 ml of 0.1M silver nitrate solution placed in a glass-stopper conical flask; dilute the solution to the 50 ml, with distilled water. Add 2 ml of Nitric acid & add 2 ml of ferric ammonium sulphate solution & titrate with 0.1M ammonium thiocyanate solutions, to the first appearance of red brown colour.

II. Assay -

Weigh accurately about 0.1 gm of Sodium chloride & dissolve in 50 ml of water in a glass-stopper flask. Add 50 ml of 0.1M Silver Nitrate solutions, 5 ml of Nitric acid, 2 ml of Di-butyl phthalate shake well & titrate with 0.1 M of Ammonium thiocyanate solutions, using 2 ml of Ferric ammonium sulphate solution as an indicator, until the colour changes to reddish brown.

EXPERIMENT NO- 5.

ASSAY of SODIUM BENZOATE with 0.1(N) of PERCHLORIC ACID

REQUIREMENTS-

Funnel, Pipette, Beaker, Volumetric flask, Burette, Sodium benzoate, Perchloric acid, glacial Acetic acid, Acetic anhydride, Crystal violet

Procedure-

I. Preparation of 0.1N Perchloric Acids -

Mixed 0.85 ml of Perchloric acids with 50 ml of glacial Acetic acid & 2.5 ml of acetic anhydride, Volume makes up to 100 ml with Acetic acid & allows the solution to stand for 24 hours.

II. Standardization of 0.1N Perchloric Acids -

Weighing accurately about 0.35 gm of Potassium hydrogen phthalate previously dried, dissolved it in 50 ml of glacial Acetic acid & add about 0.1 ml of crystal violet solutions & titrated with 0.1 N Perchloric acids until violet colour changes to emerald green colour.

Each ml of 0.1N Perchloric acid is equivalent to 0.2042 gm of Potassium hydrogen phthalate.

III. Assay of Sodium benzoate- weighing accurately about 0.25 gm of Sodium benzoate & dissolved in glacial Acetic acid, warming the solution to 50°C if necessary, cools, titrate with 0.1 M Perchloric acid, using 0.5 ml of Crystal violet solution as indicator.

EXPERIMENT NO- 6.

LIMIT TEST of CHLORIDE ION in A SUPPLIED SAMPLE of D-GLUCOSE

Requirements:

Weight box, Beaker, Glass Rod, Pipette, Chemical balance, Nessler cylinder, Silver nitrate, Nitric acid, Sodium chloride, Dextrose

Procedure-

I. Preparation of Test Solution: - Specified weight of the substance was dissolved in water & transformed to a Nessler Cylinder, & marked it as "TEST". To the solution 2 ml of Nitric acid was added & volume made up to 50 ml by addition of distilled water. Then 1 ml of (0.1M) Silver nitrate solution was added & the solution was stirred aside for 5 minutes.

II. Preparation of Standard Solution: - 10 ml 25 ppm Chloride solution was poured in a Nessler cylinder & marked it as "STANDARD". 2 ml of Nitric acid was added & the volume was made up to 50 ml by adding distilled water. Then 1 ml of (0.1M) Silver nitrate solution was mixed. Stirred with glass rod & was set aside for 5 minutes. Compare the turbidity of the test solution with the standard solution.

EXPERIMENT NO- 7.

LIMIT TEST of SULPHATE ION in A SUPPLIED SAMPLE of BORIC ACID

Requirements:

Weight box, Beaker, Glass Rod, Pipette, Chemical balance, Nessler cylinder, Boric acid, Barium chloride, Ethanolic sulphate standard solution, Acetic acid, Distilled water

Procedure-

I. Preparation of Test Solution: - 0.33 gm of Boric acid was taken & dissolved in 10 ml of boiling water in a Nessler cylinder & marked it as "TEST". The volume was adjusted to 15 ml by adding 5 ml of water. 1 ml of 25% w/v solution of Barium chloride was added to it. To this solution 1.5 ml of standard ethanolic sulphate solution was added, 0.15 ml of 5(M) Acetic acid solution was added to the above solution to just make the medium acidic. Now the final volume was adjusted up to 50 ml with distilled water. The solution was kept aside for 5 minutes.

II. Preparation of Standard Solution: - 15 ml of 10 ppm of Sulphate standard solution was taken in a Nessler cylinder & marked it as "STANDARD". 1 ml of 25% w/v solution of Barium chloride was added to it. To this solution 1.5 ml of standard ethanolic sulphate solution was added, 0.15 ml of 5(M) Acetic acid solution was added to the above solution to just make the medium acidic. Now the final volume was adjusted up to 50 ml with distilled water. The solution was kept aside for 5 minutes. Compare the opalescence of the test solution with the standard solution.

EXPERIMENT NO- 8.

LIMIT TEST of IRON in A SUPPLIED SAMLE of SODIUM BI CARBONATE

Requirements:

Weight box, Beaker, Glass Rod, Pipette, Chemical balance, Nessler cylinder, Sodium bicarbonate, Hydrochloric acid, Citric acid, Thioglycolic acid
Distilled water

Procedure –

I. Preparation of Test Solution: - 2 gm of Sodium bicarbonate samle was taken in a Nessler cylinder & marked it as a “TEST”. To this 20 ml of water was added to it. This solution was diluted by adding 10 ml of distilled water. To this above solution, 2 ml of 20%w/v solution of iron free citric acid solution was added. Now, 0.1 ml of thioglycolic acid was added. The solution was made alkaline by adding Ammonia solution. Now, the final volume was adjusted up to 50 ml. The solution was kept aside for 5 minutes.

II. Preparation of Test Solution: - 2ml of 20 ppm iron solution was taken in a Nessler cylinder & marked it as a “STANDARD”. To this 20 ml of water was added to it. This solution was diluted by adding 10 ml of distilled water. To this above solution, 2 ml of 20%w/v solution of iron free citric acid solution was added. Now, 0.1 ml of thioglycolic acid was added. The solution was made alkaline by adding Ammonia solution. Now, the final volume was adjusted up to 50 ml. The solution was kept aside for 5 minutes. Compare the pink colour of the test solution with the standard solution.