

**I SEMESTER (1<sup>st</sup> year B.Pharm)**

**PHARMACEUTICAL INORGANIC CHEMISTRY**

**PRACTICAL LAB MANUAL**

## Pharmaceutical Inorganic Chemistry Practical;

### EXPERIMENT NO- 1.

#### Limit Test for Chloride Ion in Sodium Bicarbonate

**Apparatus:** Nessler cylinder, Pipette, Glass rod.

**Chemicals required:** Silver nitrate, Nitric acid, Sodium chloride, Sodium bicarbonate, Distilled water.

#### Procedure-

Specified weight of the substance was dissolved in water & transfer to a Nessler Cylinder. To the solution 2 ml of Nitric acid was added & the volume was made up to 50 ml with water. Then 1 ml of Silver nitrate solution of 0.1M concentration was added & the solution was stirred.

Then the solution is kept aside for 5 minutes. Then the test solution was prepared in this manner. Simultaneously for the standard solution, 10ml of 25 ppm Chloride solution was poured in Nessler cylinder. 2 ml of dilute nitric acid was added & the volume was made up to 50 ml with water. Then 1 ml of 0.1M Silver nitrate solution was mixed. Stirred with a glass rod & was set aside for few minutes.

## **EXPERIMENT NO- 2.**

### **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- 3.

#### 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- 4.

### 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 sample 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.

## EXPERIMENT NO- 5.

### LIMIT TEST of IRON in A SUPPLIED SAMLE of CALCIUM CARBONATE

**Requirements:** Weight box, Beaker, Glass Rod, Pipette, Chemical balance, Nessler cylinder, Calcium carbonate, Hydrochloric acid, Citric acid, Thioglycolic acid Distilled water.

#### **Procedure –**

**I. Preparation of Test Solution:** - 0.2 gm of Calcium carbonate sample 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.

## **EXPERIMENT NO- 6.**

### **Limit Test for Arsenic in the Supplied Sample of Dextrose**

Apparatus- Gutzeit apparatus, Pipette, Measuring Cylinder

Chemicals- Dextrose, Hydrchloric acid, Potassium iodide, Zinc dust, Arsenic standard solution.

#### **Procedure-**

- I. **Preparation of Test Solution-** 10 gm of the given sample i.e. Dextrose taken in the Gutzeit apparatus. To it 50 ml of water was added, and then 10 ml of HCl was added to the above solution 5ml of 1M potassium iodide solution added. Then 10 gm of Zinc dust was added to it. Then finally the whole apparatus was assembled and kept on the water bath for few minutes.
- II. **Preparation of Standard solution-** 10 ppm of arsenic standard solution was taken in Gutzeit apparatus. 50 ml of water was added & 10 ml of HCl was added to it. Then 5 ml of 1M potassium iodide solution was added to the above solution. At last 10 gm of Zinc dust was added. Then the whole apparatus was assembled & kept for few minutes on the water bath.



## **EXPERIMENT NO- 7.**

### **Identification Test for Sodium bicarbonate**

**Procedure-** Weigh approximately 2 gm of the sample & put it in a beaker, pour about 10 ml of distilled water into the beaker & mix it until the salt dissolves completely.

Pour half of the solution in another beaker. By using a pipette add about 2 ml of HCl solution into the first beaker. If bubbles of gas ( $\text{CO}_2$ ) intensely formed then the sample is a carbonate salt (may be Sodium bicarbonate or sodium carbonate).

Cut a piece of pH paper, holding one end of the paper strip, dip the other end into the solution in the second beaker for 1-2 seconds & then take it out. The part of the paper that has been in the solution will change colour.

Compare the colour with pH scale. If the pH is around 8, the sample is  $\text{NaHCO}_3$  & if the range is in 9.5-10 it is  $\text{Na}_2\text{CO}_3$



## **EXPERIMENT NO- 8.**

### **Identification Test for Ferrous Sulphate**

#### **Requirements-**

**Apparatus-** Test-tube, Test- tube holder, Spatula

**Chemicals-** Potassium ferricyanide solution, Dilute HCl, NaOH solution, Distilled water

#### **Procedure-**

1 ml of required substance containing a minimum of 1 mg of iron was to be dissolved in 2 ml of water, to which 1ml of potassium ferricyanide solution was added which gave a dark blue precipitate that would be insoluble in dilute HCl & can be decomposed by a NaOH solution.

## **EXPERIMENT NO- 9.**

### **Preparation of Potash Alum**

#### **Requirements-**

**Chemicals-** Potassium sulphate, Aluminium sulphate, Dilute Sulphuric acid

**Apparatus-** Beaker, China dish, Funnel, Funnel stand, Glass rod, Wash bottle, Tripod stand, Wire gauze

#### **Procedure-**

- I. Take a 250ml beaker; transfer 2.5 gm of Potassium sulphate crystal to it. Added 20ml water, stir to dissolve the crystal, warm if required.
- II. Take another 250ml beaker; transfer 10gm of Aluminium sulphate crystal to it. Add 20ml of water & 1ml of dilute Sulphuric acid. Heat for about 5 minutes. If milkiness still persists filter the solution.
- III. Mix the two solutions in a china dish and place the china dish on wire gauze placed over a burner. Stir the solution with a glass rod. Concentrate the solution till the crystallization point is reached. Place the dish over a beaker containing cold water.
- IV. Soon the crystal of potash alum separate out, decant off the mother liquor and wash the crystals with a small amount of cold water.
- V. Dry the crystals by placing them between filter paper & spread them over a porous plate.