East Point College of Pharmacy

East Point Campus, Jnana Prabha, Virgo Nagar Post
Bengaluru – 560049, Karnataka

Approved
by
Pharmacy Council of India, New Delhi

Affiliated
to
Rajiv Gandhi University of Health Sciences
Karnataka
Bengaluru – 560 041
India

LAB MANUAL

PHARMACEUTICAL INORGANIC CHEMISTRY

PHARM D

1ST Year

PROGRAM SPECIFIC OUTCOMES	
DOCTOR OF PHARMACY	
PSO1	Acquire a thorough foundational knowledge in pharmaceutical sciences, including pharmacology, pharmaceutics, pharmaceutical chemistry, pharmacognosy and pharmaceutical analysis to excel in further academic pursuits
PSO2	Acquire and apply the pharmacotherapeutic concepts for better patient care enhancing employability across various sectors including clinical research organizations, academic and hospitals
PSO3	Equip with entrepreneurial skills and knowledge of pharmacoepidemiological studies and regulatory aspects to initiate and run successful ventures in the healthcare sector

Course:	**Code: 1.5P** **Pharmaceutical Inorganic Chemistry**
CO1	Capable of articulating the underlying principles governing limit tests, ion identification, purity tests, and various types of volumetric analysis, including assay principles
CO2	Utilize volumetric analysis for both quantitative estimation of drugs and mixtures
CO3	Conduct limit tests to detect impurities and perform identification tests to assess purity within the provided compounds
CO4	Acquire basic knowledge regarding general methods of preparation of inorganic compounds of pharmaceutical importance

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INTRODUCTION TO LIMIT TEST

Practically it is impossible to remove all the impurity from any substance. Some remainin trace even after purification. So it is only desirable that the substance should be sufficient pure and can be used safely.

The pharmacopoeia (IP, BP, USP) specify the limit unto which various impurity can betolerated in pharmaceutical drugs and excipients. The limit tests help to check and indicate thepresence of various impurities in substances.

The pharmacopoeia has fixed the limit of various impurities considering various factors.

Limit tests are qualitative and semi quantitative test design to detect the limits of impurity commonly present in pharmaceutical substance.

Impurity: Any foreign material present in drug or chemical is called as impurity.

Pure compound: A drug or chemical said to be pure if it is free from all impurity. **Qualitative test:** This is the by which we can identify the compound, or it is identification test.**Quantitative test:** The test by which the quantity of the substance is estimated.

In the limit test the impurity is identified and the presence of impurity is compared withthe standard taking the specified amount of impurity.

The standard substances of limit test contain the maximum amount of impurity whichcan be allowed in pharmaceutical substance.

The comparison of sample with standard involves the physical changes like colour,Turbidity or turbidity etc.

TYPES OF IMPURITY

Toxic impurity: The impurity which is very harmful and can cause death even when taken once or short period of time. Example: Arsenic

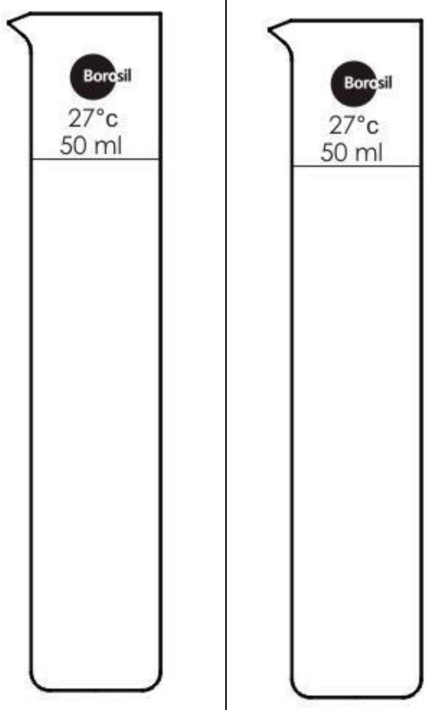
Cumulative impurity: The impurity which may not cause immediate effect, but when taken for long period of time shows toxicity. Example: Heavy metals

Harmless impurity: some impurity may not cause harm to body but if present in large quantity reduces the therapeutic activity of active ingredient. Example: Chlorides, Sulphates.

On considerations of above classification, the pharmacopoeia has fixed the permissible limit for each impurity. For toxic impurity the permissible limit is as less as 5-10 ppm. Whereas for cumulative impurity the limit is little high as 20 ppm.

Difference between assay and limit tests

Assay	Limit tests
Quantitative test	Semi-quantitative or qualitative
Results provides exact amount of substance	Results provide a range of impurities.
Test for the quantity of the substance present as well as the impurity.	Test particularly for impurity



Nessler’s Cylinder

Experiment No:01

LIMIT TEST FOR CHLORIDES

AIM: To carry out the limit test for chlorides for the given sample.

Apparatus Required: Nessler's cylinder, Glass rod, pipette.

Chemical Required: 5% silvernitrate solution, 0.05845%w/v sodium chloride, dilute nitric acid, distilled water.

Principle

The limit test for chloride is based upon the reaction between soluble chloride and silver nitrate reagent in presence of dilute nitric acid to produce insoluble silver chloride.
The turbidity produce by the sample is compared with that of standard turbidity produce by the specific amount of chloride ions.
The sample turbidity should not be greater than the standard turbidity for passing the limit test for chloride.

Reaction

Sample : $Cl^- + AgNO_3 \xrightarrow{\text{Dil } HNO_3} AgCl\downarrow + NO_3^-$

Standard : $NaCl + AgNO_3 \xrightarrow{\text{Dil } HNO_3} AgCl\downarrow + NaNO_3$

Reasons:

Dilute nitric acid: is used to prevent the interference of other impurities like Carbonates, Bicarbonates, Phosphates, and Hydroxides, which also may react with silver nitrate to give their respective precipitates.
It also increases the sensitivity of the reaction by common ion effect.
The nitrate ion of nitric acid and silver nitrate is common so the formation of precipitates of silver chloride from silver nitrate will form immediately.

Procedure

Standard Turbidity	Sample Turbidity
Pipette out 1mL of 0.05845% w/v of standard NaCl solution in a clean Nessler's cylinder. Add 10mL of dilute Nitric acid and make up the volume up to 50mL by adding distilled water. Add 1mL of 0.1M silver nitrate and allow to stand for 5min.	Dissolve the sample given in 25mL distilled water in a clean Nessler's cylinder. Add 10mL of dilute Nitric acid and make up the volume up to 50mL by adding distilled water. Add 1mL of 0.1M silver nitrate and allow to stand for 5min

Comparison

Compare the sample turbidity with standard turbidity by viewing the Nessler's cylinder against white back ground.

Observation & Report: When viewed transversely against a dark background, the turbidity produced in the sample is less / more than the standard turbidity. Hence the given sample passes / fails the limit test for Chlorides.

Question for viva and synopsis

1. Define limit test
2. What are Impurities?
3. What are Toxic impurities
4. What do you mean by pure compound?
5. Why dil. HNO_3 used in Chloride limit test?

Experiment No:02

LIMIT TEST FOR SULPHATE

AIM: To carry out the limit test for Sulphate for the given sample.

Apparatus Required: Nessler's cylinder, Glass rod, pipette.

Chemicals Required: Dilute HCl, Barium sulphate reagent, Distilled water, Standard Potassium Sulphate solution (0.1089%w/v).

Principle

The limit test for Sulphate is based upon the reaction between soluble Sulphate and Barium chloride in the form of Barium sulphate reagent in presence of Dilute HCl, it produces the turbidity due to the formation of Barium sulphate. The turbidity is compared with that of standard using 0.1089%w/v potassium sulphate
The sample Turbidity should not be greater than the standard turbidity for passing the limit test for sulphate.

Reaction-

Sample : $\mathbf{SO_4^- + BaCl_2 \xrightarrow{\text{Dil HCl}} BaSO_{4\downarrow} + 2Cl^-}$

Standard : $\mathbf{K_2SO_4 + BaCl_2 \xrightarrow{\text{Dil HCl}} BaSO_{4\downarrow} + 2KCl}$

Composition & Preparation of Barium Sulphate Reagent

1. 15mL of 0.5M Barium Chloride used as precipitating agent.
2. 5mL of 0.01089%w/v potassium sulphate used as seeding agent and there by increased sensitivity of the reaction
3. 20 mL of sulphate free alcohol is used to avoid super-saturation of Barium sulphate.
4. Make up the volume to 50mL with distilled water.

Reasons

Dilute HCl: is used to prevent the interference of other impurities like Carbonates, Chloride Bicarbonates, Phosphates, and Hydroxides which also reacts with Barium chloride to give their respective precipitates and also the other precipitates are soluble in dilute HCl.
HCl acid also increases the sensitivity of the reaction by common ion effect.
The Chloride ion of HCl and Barium chloride is common so the formation of precipitates of Barium sulphate from Barium Chloride will produce fast.

Procedure

- To 1.0 mL of 25.0 per cent w/v solution of *barium chloride* in a Nessler's cylinder add 1.5 mL of standard sulphate solution (10 ppm SO_4), mix and allow to stand for 1 minute.
- Add 15 mL of the solution prepared as directed in the monograph or a solution of the specified quantity of the substance under examination in 15 mL of *water* and 0.15 mL of 5 M acetic acid.
- Add sufficient *water* to produce 50 mL,
- Stir immediately with a glass rod and allow to stand for 5 minutes.
- When viewed transversely against a dark background any turbidity produced is not more intense than that obtained by the standard.

Standard Turbidity	Sample Turbidity
Pipette Out 1 mL of 0.1089% w/v of standard potassium sulphate solution in a clean Nessler's cylinder and dilute to 25 mL with distilled water. Add 2 mL of dilute HCl and 5 mL of Barium Sulphate reagent. Make up the volume up to 50 mL by adding distilled water. Allow to stand for 5min	Dissolve the specified amount of sample with distilled water in a clean Nessler's cylinder. Add 2 mL of dilute HCl and 5 mL of Barium Sulphate reagent. Make up the volume up to 50 mL by adding distilled water. Allow to stand for 5min

Comparison

Compare the sample turbidity with standard turbidity by viewing the Nessler's cylinder against white back ground.

Observation & Report: When viewed transversely against a dark background, the turbidity produced in the sample is less / more than the standard turbidity. Hence the given sample passes / fails the limit test for Sulphates.

Questions for viva and synopsis

1. Explain the principle involved in Sulphate limit test.
2. Write the composition of Barium sulphate reagent. Explain their use.
3. Why Alcohol is used in Sulphate limit test (in Barium Sulphate reagent).

Experiment No:03

LIMIT TEST FOR IRON

AIM: To carry out the limit test for Iron for given sample.

Apparatus Required: Nessler's cylinder, Glass rod, pipette.

Chemical Required: 20%w/v citric acid, Thioglycolic acid, Dil. Ammonia Solution/Ammonium Hydroxide solution, Litmus paper, distilled water and standard ferric ammonium sulphate solution (0.0173 gm in 1000 mL of water).

Principle

The limit test for Iron is based upon the reaction between ferrous ion and Thioglycolic acid to produce pale pink color to reddish violet color, due to the formation of ferrous thioglycolate complex in ammonia solution.

The oxidation state of Iron is not important because the thioglycolic acid is a strong reducing agent which reduce the Ferric ion to ferrous ion.

The color produce by the sample is compared with that of the Standard color produced by specific amount of ferric ammonium sulphate solution under the same reaction condition.

For the sample to pass the limit test for Iron, the sample color should not be more than that of standard colour.

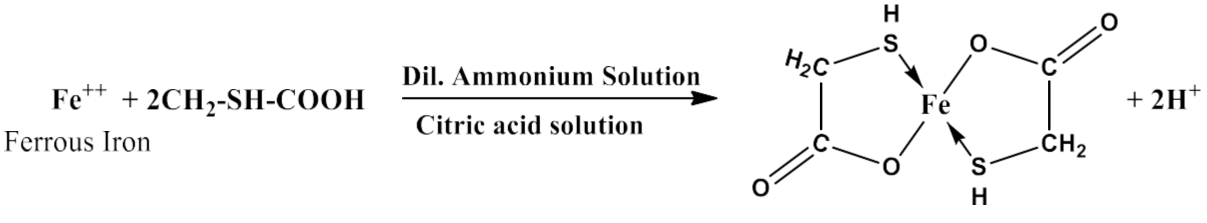
Reaction

Sample :

$$2Fe^{+++} + 2CH_2\text{-}SH\text{-}COOH \xrightarrow[\text{Citric acid solution}]{\text{Dil. Ammonium Solution}} 2Fe^{++} + HCOO\text{-}CH_2\text{-}S\text{-}S\text{-}CH_2\text{-}COOH + 2H^{+}$$

Ferric Iron Thioglycollic Acid Ferrous Iron

Standard :



Ferrous Thioglycolate Complex

Reasons

- Citric acid solution is added because it forms a soluble complex with iron and avoids precipitation as its hydroxide when Dil. ammonia solution is added.
- Dil. Ammonia solution is added to make the solution alkaline because ferrous thioglycolate gives pink color only in basic media but remain color less in acidic or neutral media.

Procedure

Standard Colour	Sample Colour
Pipette out 2 mL of standard ferric ammonium sulphate solution in to a clean Nessler's cylinder and dilute to 25 mL with distilled water. Add 2 mL of 20%w/v citric acid and 0.1 mL thioglycolic acid. Make up the solution alkaline by adding dil. ammonia solution and check with Litmus paper. Dilute to 50 mL with distilled water, mixed and allowed to stand for 5 min.	Dissolve the specified amount of sample with 25 mL distilled water in a clean Nessler's cylinder. Add 2 mL of 20%w/v citric acid and 0.1 mL thioglycolic acid. Make up the solution alkaline by adding dil. Ammonia solution and check with Litmus paper. Dilute to 50 mL with distilled water, mixed and allowed to stand for 5 min.

Preparation of standard ferric ammonium sulphate solution:
Dissolve 0.173g of Ferric ammonium sulphate $[NH_4Fe(SO_4)_2]$, $2H_2O$ to 1.5 mL HCl. Add sufficient water to make up the volume 1000 mL.

Comparison

Compare the sample color with standard color by viewing the Nessler's cylinder against white back ground.

Observation & Report: When viewed transversely against a white background, the color produced in the sample is less / more than the standard color. Hence the given sample passes / fails the limit test for Iron.

Questions for viva and synopsis

1. Explain the principle involved in Iron limit test
2. Write the use of dil. Ammonia solution and citric acid in iron limit test.
3. Write the use of Thioglycolic acid is acid in iron limit test.
4. Name the standard substance used in iron limit test.

Experiment No:04

LIMIT TEST FOR HEAVY METALS

AIM: To carry out the limit test for Heavy metal for the given sample.

Apparatus Required: Nessler's cylinder, Glass rod, pipette.

Chemical Required: Hydrogensulphide solution, sodium sulphide.

Principle

The limit test for heavy metal is based upon the reaction between heavy metal ion and hydrogen sulphide solution(freshly prepared) or sodium sulphide to produce black or brown color precipitate of heavy metal sulphide at suitable pH around 3-4 which could be maintained by adding dilute acetic acid or ammonia..

The color produce by the sample is compared with that of Standard color produced by specific amount of lead nitrate solution under the same reaction condition.

For the sample to pass the limit test for heavy metal the sample color should not be more than that of standard colour.

Procedure

Method A-

This method is applied for those substances which gives clear colorless solution under the specified condition**.**

Standard Colour	Sample Colour
Pipette out 2 mL of lead nitrate solution in to a clean Nessler's cylinder and dilute to 25 mL with distilled water.	Dissolve the specified amount of sample with 25 mL distilled water in a clean Nessler's cylinder.
The pH. of the solution is adjusted between 3-4 by adding dilute acetic acid and ammonia.	The pH. of the solution is adjusted between 3-4 by adding dilute acetic acid and ammonia.
Make up the volume to about 35 mL by adding water.	Make up the volume to about 35 mL by adding water.
Add 10 mL freshly prepared hydrogen sulphide solution and make up the volume to 50 mL with distilled water.	Add 10 mL freshly prepared hydrogen sulphide solution and make up the volume to 50 mL with distilled water.

Reason:

- The pH is maintained 3-4 because in this pH range the heavy metal sulphide precipitate is more stable.
- The hydrogen sulphide solution is freshly prepared because on keeping the hydrogen sulphide gas escape into atmosphere.

Method B-

This method is applied for those substances which does not give clear colorless solution under the specified condition as per method A.

The sample solution has to be made in a different way but the standard solution is same as method A.

Preparation of sample solution for method B

1. Specified quantities of sample as per monograph are taken in a crucible.
2. Moistened with Sulphuric acid and ignited until charred.
3. A few drops of nitric acid is added and the mixture is heated about 500 ^oC.
4. It is then allowed to cool and the residue is digested with 10 mL of Dil. HCl for 2-3 min.
5. The excess acid is neutralized by NH_3 solution and dilute with water and filtered.
6. 35 mL of the above solution is taken in a Nessler's cylinders. Add 10 mL of freshly prepared H_2S solution and make up the volume with water up to 50 mL.

Reason:
Heating the sample with acids is done to remove non-metallic substances which will interfere with the limit test

Method C-
It is for those substances which gives clean colorless solution in NaOH

Standard Colour	Sample Colour
Pipette out 2 mL of lead nitrate solution in to a clean Nessler's cylinder. The pH. of the solution is adjusted between 3-4 by adding Add 5 mL 10% NaOH and 5 drops of Na_2S solution and make up the volume to 50 mL with distilled water.	Dissolve the specified amount of sample with 25 mL distilled water in a clean Nessler's cylinder. Add 5 mL 10% NaOH and 5 drops of Na_2S solution and make up the volume to 50 mL with distilled water.

Comparison

Compare the sample color with standard color by viewing the Nessler's cylinder against white back ground.

Observation & Report: When viewed transversely against a dark background, the color produced in the sample is less / more than the standard Color. Hence the given sample passes / fails the limit test for Heavy Metals.

Question for viva and synopsis
1. Explain the principle involved in Heavy metals limit test.
2. Why dil. ammonia solution and citric acid solution are used in Heavy metals limit test?
3. Why Hydrogen sulphide solution has to prepare freshly in Heavy metals limit test?
4. When Na_2S and H_2S are used as reagent in Heavy metals limit test?

Experiment No:05

LIMIT TEST FOR LEAD

AIM: To carry out the limit test for lead for the given sample.

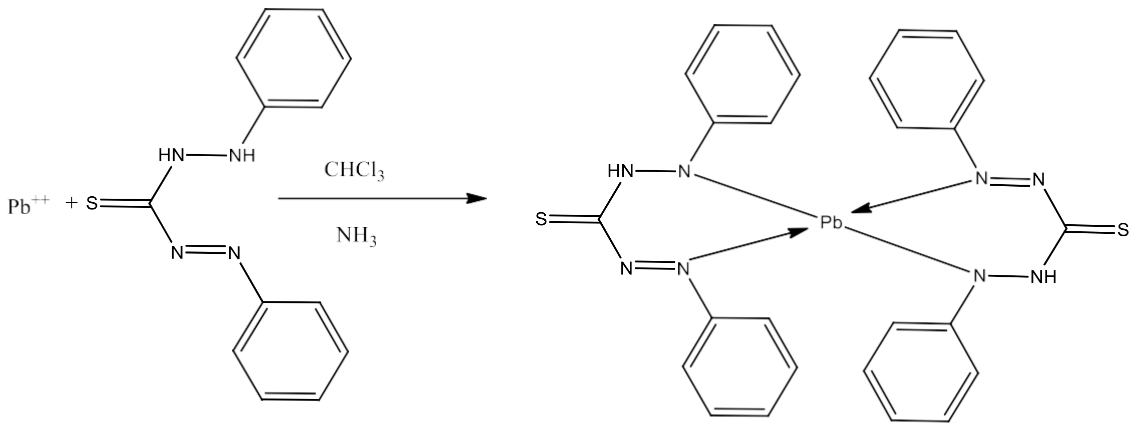
Apparatus Required: Beaker, Glass rod, pipette.

Chemical Required: ammonium citrate, hydroxyl amine hydrochloride, phenol red, dil. Ammonia solution, potassium cyanide, Dithizone solution in chloroform, and standard lead nitrate.

Principle

The limit test for lead is based upon the reaction between lead and diphenyl thiocarbazone or Dithiazone in chloroform is able to extract lead impurities from alkaline aqueous solution as a lead Dithizone complex which is red in color. The original Dithizone is having green color in chloroform while the lead Dithizone having violet or the red color. The intensity of color complex is depending upon on the amount of lead in solution. The color of lead Dithizone complex in chloroform is compared with the color produced by standard lead nitrate solution treated in a same manner.

Reaction-



Procedure

For sample-

A specified amount of sample solution is prepared as directed in IP and is taken in a separating funnel. 6 mL of ammonium citrate, 2 mL hydroxyl amine hydrochloride, 2 drops of phenol red is added. The solution is made alkaline by adding dil. ammonia solution and 2 mL of potassium cyanide is added. The alkaline solution is extracted with 5 mL portion of Dithizone solution in chloroform. Extraction is continued until the color of Dithizone layer remains green. The combined chloroform extract is shaken with 1% nitric acid, the Dithizone layer is taken into a beaker.

For standard- Specified quantity of lead nitrate is treated in the same manner as the sample solution

Reasons:

- Reagent like hydroxyl amine hydrochloride, KCN, are added to prevent the interference of other impurities.
- Dil. Ammonia solution is added to make the solution alkaline which will be indicated by phenol red indicator, at this pH the extraction is optimum.

Comparison

Compare the sample color with standard color by viewing the Nessler's cylinder against white back ground.

Observation & Report: When viewed transversely against a dark background, the turbidity produced in the sample is less / more than the standard turbidity. Hence the given sample passes / fails the limit test for Lead.

Question for viva and synopsis

1. Why the reagent like hydroxyl amine hydrochloride, KCN are added to lead limit test?
2. Explain the principle and reaction in lead limit test?
3. Write Dithizone test

Experiment No:06

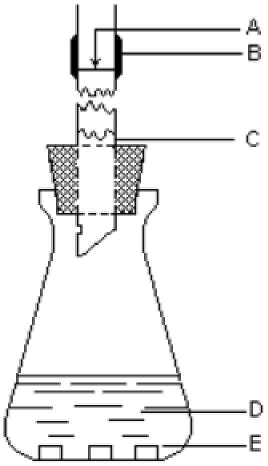
LIMIT TEST FOR ARSENIC

AIM: To carry out the limit test for arsenic for the given sample.

Gutzeit's Apparatus :

It consists of a wide mouthed glass bottle of 120 mL capacity fitted with rubber bung through which a delivery tube of 20 cm length is placed having external diameter 8 mm and internal diameter of 6.5 mm. The tube is constricted at lower end with a hole of 2 mm diameter and also a side hole of 1mm diameter. The upper end of the tube is fixed with two rubber bung sucha way that a mercuric chloride paper is sandwiched in between them. The two rubber bung areheld together tightly with a clip or a screw clamp. Inside the tube, 2 mm below the rubber bunglead acetate cotton is placed.

APPARATUS FOR LIMIT TEST FOR ARSENIC



A. Mercuric chloride test paper disc
B. Rubber connexion
C. Cotton gauze saturated with lead acetate
D. Test solution
E. Aluminum squares

Principle

The limit test for arsenic is based upon the conversion of arsenic impurity by a series of reaction to arsine gas which react with mercuric chloride test paper to give yellow or brown color strain of mercuric arsenide.

The stain produce by the sample is compared to the standard stain produce from specific amount of arsenic trioxide under the same reaction condition.

For the sample to pass the limit test of arsenic the sample stain should not be more than that of standard stain.

Reactions

1. Trivalent or pentavalent arsenic impurity is converting to arsenous acid and arsenic acid by dilute HCl.

$$As^{+++} \xrightarrow{\text{Dil HCl}} H_3AsO_3$$

$$As^{+++++} \xrightarrow{\text{Dil HCl}} H_3AsO_4$$

2. the arsenic acid formed is reduce to arsenous acid by reacting with stannous chloride , HCl and KI

$$H_3AsO_4 \xrightarrow{\text{HCl / KI / SnCl}_2} H_3AsO_3$$

3. Arsenous acid is further reduce to arsine gas by reacting with nascent hydrogen which is obtained from zinc and HCl

$$Zn + 2\,HCl \longrightarrow ZnCl_2 + 2[H]$$

$$H_3AsO_3 + 6\,[H] \longrightarrow AsH_3\uparrow + 3H_2O$$

4. Arsine gas react with mercuric chloride test paper to give yellow or brown stain of mercuric arsenide

$$AsH_3\uparrow + HgCl_2 \longrightarrow Hg\,(AsH_2)_2 + 2HCl$$

Preparation of the apparatus for the arsenic limit test

1. The glass bottle (Gutzeit Apparatus) and the tube are first washed with dilute HCl and rinsed with water.
2. The delivery tube is then tightly packed with lead acetate cotton.
3. The upper end of the tube is inserted into a rubber bung on which a square piece of mercuric chloride test paper is placed.
4. The second bung is placed over this and the two bungs are tightly attached with spring clip.
5. The lower end of the delivery tube is inserted into another rubber bung.

Procedure for standard

Place 50 mL of water in wide mouth bottle. Add 10 mL of standard HCl, 1g of KI. Pipette out 10 mL of standard Arsenic trioxide and transfer to the bottle, add 10g of granulated Zinc and quickly place the prepare glass tube in its position. Allow to stand for 40 min at 40 ^{o}C.

Procedure for sample

Place 50 mL of water in wide mouth bottle, add 10 mL of standard HCl, 1g of KI and specified amount of sample in to the bottle. Add 10g of granulated Zinc and quickly place the prepare glass tube in its position. Allow to stand for 40 min at 40 ^{o}C.

Reagent and chemicals required

All the reagents and chemicals used arsenic limit test must be completely free from arsenic and labelled as **AsT** except the sample and standard solution.

Mercuric Chloride Test Paper

It is smooth white filter paper, not less than 25mm in width, which is first shocked in saturated solution of $HgCl_2$ and finally dried at 60 ^{o}C in dark.

Precautions

1. The apparatus must be properly washed with dilute HCl and the rinsed with water every time before and after the Arsenic limit test.
2. Mercuric chloride test paper must be freshly prepared by wetting the test paper in Mercuric chloride solution and drying under shade.
3. The delivery tube must not dip in to the mixture in the bottle, so that the Arsine gas allowed passing through it.
4. The reaction must be allowed to proceed at 40 ^{0}C for 40min.
5. The standard strain and the sample strain should be prepare simultaneously and compare immediately after the reaction.
6. Lead acetate cotton plug should be keep freshly prepared.

Reasons

1. Stannous chloride is added because it acts as a reducing agent in presence of HCl. It converts arsenic acid to arsenous acid.

2. Granulated zinc is added to produce the nascent hydrogen gas by reacting with HCl. The nascent hydrogen gas is more reactive which convert arsenous acid to arsine gas.

3. Granulated zinc contains an impurity of sulphur which reacts with HCl and produce H_2S gas. So lead acetate cotton is placed in the delivery tube to trap the H_2S gas by reacting with them, which otherwise react with $HgCl_2$ paper and produce black color and the stain produce by arsine gas will not be visible.

$$H_2S \quad + \quad HgCl_2 \quad \text{------------------->} \quad HgS \quad + \quad 2HCl$$

$$H_2S \quad + \quad Pb\,(CH_3COO)_2 \quad \text{------------------>} \quad \underset{\text{Black}}{PbS} \quad + \quad CH_3COOH$$

4. A side whole is provided in the delivery tube to allow continuous passage for arsine gas in the case where the lower whole blocked due to condensation of water vapor.

5. The stains are immediately compared because they fade on exposure to light. They can be preserved by dipping the test paper in molten paraffin wax and drying.

Report

The color intensity formed in the sample mercuric chloride paper was found to be more/less than the color intensity formed in the standard mercuric chloride paper. Hence the given sample fails/passes the limit test for Arsenic.

Questions the for viva and synopsis

1. Explain principle involved in Arsenic limit test.
2. Draw a neat labeled diagram of Gutzeit apparatus.
3. Why KI, Zn and HCl used in arsenic limit test
4. Why side hole made in the delivery tube in Gutzeit apparatus
5. Explain why lead acetate cotton is used in arsenic limit test.
6. How to preserve the stain on test paper
7. Why Mercuric chloride test paper has to prepare freshly?

Experiment No:07

SPECIAL PROCEDURE FOR LIMIT TEST OF CHLORIDE AND SULPHATE FOR SODIUM BICARBONATE

AIM: To perform a special procedure in limit test of sulphate and chloride in sodiumbicarbonate.

Apparatus Required: Nessler's cylinder, Glass rod, pipette.

Chemical Required: 5% silvernitrate solution, 0.05845%w/v sodium chloride, dilute nitric acid, Dilute HCl, Barium sulphate reagent, Distilled water, Standard Potassium Sulphate solution (0.1089%w/v).

Principle

In this special procedure the acid is added to neutralize the alkaline carbonate and then the sulphate ion reacts with barium chloride as barium sulphate reagent.

Reaction

$$CO_3^- + 2HCl \rightarrow H_2O + CO_2 + H^+$$

Procedure

For sulphate

Suspend 1g of sample in 10 mL of distilled water, neutralize with HCl and dilute to 15 mL with distilled water

Standard Turbidity	Sample Turbidity
Pipette out 1 mL of 0.1089% w/v of standard potassium sulphate solution in a clean Nessler's cylinder and dilute to 25 mL with distilled water Add 2 mL of dilute HCl and 5 mL of Barium Sulphate reagent. Make up the volume up to 50 mL by adding distilled water. Allow to stand for 5min	Take above solution in a clean Nessler's cylinder. Add 2 mL of dilute HCl and 5 mL of Barium Sulphate reagent. Make up the volume up to 50 mL by adding distilled water. Allow to stand for 5min

Comparison

Compare the sample Turbidity with standard Turbidity by viewing the Nessler's cylinder against white back ground.

For chloride

1.25g in dissolve in 15 mL of water add 2 mL of nitric acid

Standard Turbidity	Sample Turbidity
Pipette out 1 mL of 0.05845% w/v of standard NaCl solution in a clean Nessler's cylinder. Add 10 mL of dilute Nitric acid and make up the volume up to 50 mL by adding distilled water. Add 1 mL of 0.1M silver nitrate and allow to stand for 5min	Above solution is taken in a clean Nessler's cylinder. Add of dilute Nitric acid. Add 1 mL of 0.1M silver nitrate and allow to stand for 5min

Comparison

Compare the sample turbidity with standard turbidity by viewing the Nessler's cylinder against white back ground.

Report

1. When viewed transversely against a dark background, the turbidity produced in the sample ($KMnO_4$) is less / more than the standard turbidity. Hence the given sample passes / fails the limit test for Chloride.
2. When viewed transversely against a dark background, the turbidity produced in the sample ($KMnO_4$) is less / more than the standard turbidity. Hence the given sample passes / fails the limit test for Sulphate.

Question for viva and synopsis

1. Why HCl is added to the sample before doing the limit test?

INTRODUCTION TO VOLUMETRIC ANALYSIS

Analytical chemistry is a branch of chemistry which deals with the analysis of substances. There are two types of analysis named quantitative and qualitative analysis.

Qualitative analysis- is primarily concerned with the identification of constituents present in a chemical substance or a mixture of substances.

Quantitative analysis-is primarily concerned with the determination of exact quantity of constituent present in a given chemical sample.
Volumetric analysis is a mode of quantitative analysis which is based on the determination of volume of a solution of known concentration (standard) required to react quantitatively with a solution of a substance to be analyzed.
Volumetric analysis is also called as titrimetric analysis.

Titration: Titration is a process by which the concentration of an unknown substance in solution is determined by adding measured amounts of a standard solution that reacts with the unknown. Then the concentration of the unknown can be calculated using the stoichiometry of the reaction and the number of moles of standard solution needed to reach the so-called endpoint

Titrant: The substance of known concentration which is used to determine the unknown concentration of substance

Titrand/titrate: The substance whose concentration is not known.

Indicator: The external substance added to the titration which indicates the completion of there action by visual change is called as indicator.

End point: The point at which the reaction between titrant and Titrand is completed and the indicator change its colour to indicate the completion of these action is called as end point

Equivalent point: The exact point at which the reaction between titrant and Titrand is completed before the colour change of indicator

Titration error: little difference between endpoint and equivalent point is called as titration error.

Standard solution

The solution of a substance having known concentration and purity which is used to determine the concentration or amount of unknown substance.

Standardization is a process to find out the exact strength of some solution using some standard solution.

There are two types of standard solution that is primary and secondary standard.

PRIMARY STANDARD: This is a chemical having maximum purity and whose standard solution can be prepare exactly by weighing required quantity of substance and dissolve insufficient amount of solvent. The solution of standard substance made need not to be standardized. This solution is used for standardization of other solution.

Properties of primary standard

- It should be available with maximum purity.
- It should weigh exactly and should not absorb moisture or CO_2 while weighing.
- It should be stable and remain its chemical composition during use.
- The total amount of impurities should be in limit generally should not exceed0.01-0.02%
- It should have high molecular weight to avoid weighing error.
- It should dissolve with solvent freely

Example

For Acid base neutralization titration -Oxalic acid, Na_2CO_3, succinic acid, Benzoic acid

For Non-aqueous titration-Potassium hydrogen phthalate

For Complexometric Titration-Lead nitrate

For Precipitation titration- NaCl, Silver nitrate, potassium dichromate.

For Redox Titration-Oxalic acid, potassium dichromate

SECONDARY STANDARD:

This is a substance whose standard solution cannot be made exactly just by weighing required quantity of substance and dissolve insufficient amount of solvent. The solution made has to be standardize with the help of primary standard.

- Examples:
  - Acid base neutralization titration –HCl, Sulphuric acid, NaOH
  - Non-aqueous titration –Perchloric acid
  - Complexometric titration –Na_2¬EDTA
  - Precipitation titration –Ammonium thiocyanate,
  - Redox titration –Potassium per manganate, Sodium thiosulphate.

END POINT DETECTION:

Measuring the endpoint of a titration

Different methods to determine the end point include:

- pH indicator:
- A potentiometer
- pH meter
- Conductance
- Colour change
- Precipitation
- The rate of temperature change.
- Spectroscopy
- Amperometry

METHODEXPRESSINGCONCENTRATION

NORMALITY: It is the number of gram equivalent of the solute that present in one-liter of solution.

MOLARITY: It is the number of gram mole of the solute that present in one liter of solution.

MOLALITY: It is the number of gram mole of the solute that present in1000g of solution.

%W/V: It is the number of gram of solutepresentin100mL of solution.

%W/W: It is the number of gram of solute present in100g of solution.

%V/V: It is the number of mL of solute present in 100mL of solution.

%V/W: It is the number of mL of solute present in 100gofsolution.

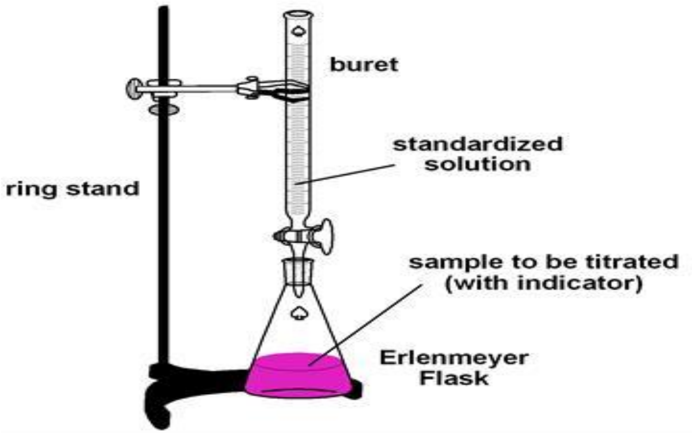
TYPES OF TITRATIONS

I. Based upon the Principle of reaction

1. Acid base neutralization titration
    - (a) Acidimetry
    - (b) Alkalimetry
    - (c) Non-aqueous titration
2. Complexometric titration
3. Precipitation titration
    - (a) Mohr’s method
    - (b) Volhard method
    - (c) Fajan’smethod
4. Redox titration
    - (a) Permanganometry
    - (b) Dichrometry
    - (c) Cerimetry
    - (d) Titanometry
    - (e) Titration with Iodine
        1. Iodometry
        2. Iodimetry
    - (f) Titration with $KBrO_3$
    - (g) Titrationwith2,4-dichlorophenolindophenol

II. Based upon the Method

1. Direct
2. Back
3. Modified/indirect
4. Replacement



Experiment No:08

ASSAY OF AMMONIUM CHLORIDE

AIM: Carry out the assay of Ammonium chloride by modified acid base titration

Apparatus Required: Burette, Pipette, Conical Flask, Volumetric Flask
Chemicals Required: 0.1N NaOH, 0.1N Oxalic acid, phenolphthalein indicator and previously neutralize formaldehyde solution.

Principle; Modified Acid base Back titration / Formalin method

The ammonium chloride is titrated by modified acid base titration where the formaldehyde react with ammonium chloride and convert them to Hexamethylene tetramine (Hexamine) and liberate equivalent amount of HCl. The liberated HCl is titrated against standard NaOH using phenolphthalein indicator. The end point color change will be the colorless to pale pink/pink.

$$4NH_4Cl \quad + \quad 6HCHO \quad \longrightarrow \quad 4HCl + 6\,H_2O + (CH_2)_6N_4HCl$$

$$HCl + 2NaOH \quad \longrightarrow \quad NaCl \quad + \quad H_2O$$

NOTE: The formaldehyde solution should be previously neutralized with 0.1N NaOH by adding phenolphthalein indicator because on auto oxidation of formaldehyde liberates formic acid during storage which will interfere the titration and gives the error in burette reading.

Procedure

PREPARATION AND STANDARDIZATION OF 0.1N NaOH

- Dissolve around 0.4g of NaOH in distilled water and make up the volume 100 mL by adding water which will produce approximately 0.1N NaOH.
- 0.63g of oxalic acid are dissolved in water and make up the volume 100 mL by adding water which will produce 0.1N oxalic acid.
- Pipette out 10 mL of 0.1N oxalic acid in a clean conical flask add 10nl of water and add 2 drops of phenolphthalein indicator.
- Titrate the solution against prepared 0.1N NaOH until the appearance of pink color.
- Repeat the procedure take the average reading and calculate the exact normality of NaOH by using the formula $N_1V_{1\,(NaOH)} = N_2V_{2\,(Oxalic\ acid)}$

ASSAY OF AMMONIUM CHLORIDE

- Dissolve the given sample in volumetric flask by adding water. Pipette out 20 mL of the solution in a clean conical flask,
- Add 20 mL of previously neutralize formaldehyde solution and 2 drops of phenolphthalein indicator.
- Titrate the solution against standard 0.1N NaOH until the appearance of pale pink color.

- Repeat the procedure take the average reading and calculate the amount of sample present by using following formula.

Observation and Calculation:

Standardization of 0.1N Sodium Hydroxide

Burette : 0.1 N NaOH
Conical Flask : 10 mL 0.1N Oxalic acid + 2 drops Indicator
Indicator : Phenolphthalein
End Point : Colorless to pale pink

Oxalic acid Vs 0.1N Sodium Hydroxide

Sl No.	Volume of Oxalic acid (mL)	Volume of NaOH(0.1N)		Volume of NaOH consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

Calculation:

$$N_1V_{1\ (\text{NaOH})} = N_2V_{2\ (\text{Oxalic acid})}$$

$$N_1 = \frac{N_2V_2}{V_1}$$

Assay of Ammonium Chloride

Burette : 20 mL Sample
Conical Flask : 20 mL previously neutralized Formaldehyde (Neutralized with phenolphthalein)
Indicator : Phenolphthalein
End Point : Colorless to pale pink

Oxalic acid Vs 0.1N Sodium Hydroxide

Sl No.	Volume of Sample (mL)	Volume of NaOH (0.1N)		Volume of NaOH consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

Calculation:

IP FACTOR
Each mL of 0.1N NaOH is equivalent to 0.005349g of NH_4Cl
Amount of sample present in 100 mL of the solution =

$$\frac{\text{Burette reading} \times \text{IP factor} \times \text{Actual normality of titrant} \times 100}{\text{IP normality} \times \text{volume of sample taken for titration}}$$

Report:

The percentage purity of the given sample of ammonium chloride was found to be ______%.

Question for viva and synopsis

1. Explain the principle involved in the assay of Ammonium chloride
2. Explain Modified titration
3. How to prepare0.1N NaOH?
4. How to standardize0.1N NaOH?
5. Why neutral HCHO is added to the titration
6. How to prepare neutralized formaldehyde.
7. Write the structure of Hexamethylenetetramine
8. How to calculate IP Factor.
9. Why neutral HCHO cannot be store for longtime?

Experiment No: 09

ASSAY OF FERROUS SULPHATE

AIM: Carry out the assay of ferrous sulphate by redox Cerimetry titration

Apparatus Required: Burette, Pipette, Conical Flask, Volumetric Flask

Chemicals Required: 0.1N ceric ammonium sulphate (CAS), 0.1N ferrous ammonium sulphate (FAS), dilute Sulphuric acid and ferroin sulphate as indicator.

Principle:

Redox Cerimetry Titration

CAS is a strong and stable oxidizing agent than $KMnO_4$ and $K_2Cr_2O_7$. It has high oxidizing potential 1.45. It is bright yellow in color and can be used as self-indicator but suitable indicator is added for clear end point.

The sample dissolve in water and add dilute Sulphuric acid and the above solution is titrated against standard ceric ammonium sulphate using ferroin sulphate solution as indicator which change red color to light blue color at the end point

Advantage of Cerimetry titration over Permanganometry Titration

1. The preparation of standard solution is easy.
2. The CAS is heat stable so we can apply heat for solubilizing the CAS in water
3. Solubility of CAS in water is high than $KMnO_4$
4. It produces transparent solution.
5. CAS has adequate oxidation potential (1.45) to oxidize other.
6. The CAS has high molecular weight so the preparation of standard solution is easy.
7. CAS can also be used as self-indicator like $KMnO_4$.

Procedure

Preparation & Standardization of 0.1N CAS

- Dissolve 6.23g of CAS in 3 mL of Sulphuric acid make up the volume 100 mL by adding distilled water which will produce approximately 0.1N CAS.
- 3.92g of FAS are dissolved in 4 mL of Sulphuric acid and make up the volume 100 mL by adding distilled water which will produce 0.1N FAS.
- Pipette out 10 mL of 0.1N FAS in a clean conical flask add 2 drops of ferroin sulphate indicator.
- Titrate the solution against prepared 0.1N CAS until the color change from red to light blue.
- Repeat the procedure take the average reading and calculate the exact normality of CAS by using the formula $N_1V_{1(CAS)} = N_2V_{2\,(FAS)}$

Assay of Ferrous Sulphate

- Dissolve the given sample in volumetric flask by adding distilled water.
- Pipette out 20 mL of the solution in a clean conical flask, add 20 mL of Sulphuric acid and 2 drops of ferroin sulphate indicator.
- Titrate the solution against prepared 0.1N ceric ammonium sulphate until the color change from red to light blue.

- Repeat the procedure take the average reading and calculate the amount of sample present by using following formula.

Observation and Calculation:

Standardization of 0.1N CAS

Burette : 0.1 N CAS
Conical Flask : 10 mL 0.1N FAS + 2 drops Indicator
Indicator : Ferroin Sulphate
End Point : Red to light blue

CAS Vs 0.1N FAS

Sl No.	Volume of FAS (mL)	Volume of CAS (0.1N)		Volume of CAS consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

Calculation:

$$N_1V_{1\,(CAS)} = N_2V_{2\,(FAS)}$$

$$N_1 = \frac{N_2V_2}{V_1}$$

Assay of Ferrous Sulphate

Burette : CAS
Conical Flask : 20 mL Sample + 20 mL sulphuric acid+ 2 drops indicator
Indicator : Ferroin Sulphate
End Point : Red to light blue

0.1N CAS Vs Sample

Sl No.	Volume of Sample (mL)	Volume of CAS (0.1N)		Volume of CAS consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

Calculation:

IP FACTOR
Each mL of 0.1N ceric ammonium sulphate is equivalent to 0.02789g of $FeSO_4$

Amount of sample present in 100 mL of the solution =

$$\frac{\text{Burette reading} \times \text{IP factor} \times \text{Actual normality of titrant} \times 100}{\text{IP normality} \times \text{volume of sample taken for titration}}$$

Report
The percentage purity of ferrous sulphate was found to be____________%

Question for viva and synopsis

1. Explain the principle involved in assay of ferrous sulphate
2. How to standardize Ceric Ammonium Sulphate?
3. What are the Advantage of Cerimetry titration over Parmanganometry Titration?

Experiment No: 10

ASSAY OF COPPER SULPHATE

AIM: Carry out the assay of Copper Sulphate by Redox Iodometry titration

Reference: Indian Pharmacopoeia vol I, 2007, pg 313.

Apparatus Required: Burette, Pipette, iodine Flask, Volumetric Flask

Chemicals Required: 0.1N sodium thiosulphate, 0.1N potassium dichromate, KI, Dilute acetic acid, potassium thiocyanate and starch mucilage solution as an indicator.

Principle: Redox Iodometry titration.

The copper sulphate first reacts with potassium iodide to yield cupric iodide and K_2SO_4. The cupric iodide is unstable which immediately liberates iodine and cuprous iodide. The liberated iodine is titrated against standard sodium thiosulphate using starch mucilage as an indicator towards the end point. The end point color will be blue to color less.

$$CuSO_4 + 4KI \longrightarrow 2CuI_2 + 2K_2SO_4$$

$$2CuI_2 \longleftrightarrow Cu_2I_2 + I_2$$

$$I_2 + Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$$

NOTE:

1. The conversion of cupric iodide to cuprous iodide and iodine is reversible, so it is necessary to make the reaction irreversible by adding potassium thiocyanate. Otherwise, the liberated iodine again reacts with cuprous iodide and form cupric iodide. The potassium thiocyanate added, react with cuprous iodide and produce cuprous thiocyanate.

2. The starch mucilage indicator should be used towards the end point because if it is used at the beginning it forms a stable complex with iodine and the iodine will not be free for reacting with sodium thiosulphate.
3. Keep the reaction mixture in dark place for 5min because the reaction is photosensitive and it needs little time to react.

Procedure

Preparation & Standardization of 0.1N $Na_2S_2O_3$

0.1N $Na_2S_2O_3$ can be prepare by dissolving around 25g of $Na_2S_2O_3$ in distilled water add 100mg of sodium carbonate and make up the volume 1000 mL by adding distilled water add 2 drops of chloroform. The solution is standardized by 0.1N $K_2Cr_2O_7$ solution which will be prepare by dissolving 4.9g $K_2Cr_2O_7$ in distilled water and make up the volume 1000 mL by adding distilled water.

- Pipette out 10 mL of 0.1N $K_2Cr_2O_7$ in a clean iodine flask add 10 mL of water, 2 mL of Con HCl and add 10 mL of 15% KI solution.
- Keep the reaction mixture in a dark place for 5min.
- Titrate the liberated iodine against prepared 0.1N $Na_2S_2O_3$ until the appearance of straw yellow color.
- Add 1 mL of starch mucilage solution and continue the titration until disappearance of dark blue color.
- Repeat the procedure take the average reading and calculate the exact normality of $Na_2S_2O_3$ by using the formula N_1V_1 (Sodium thiosulphate) = N_2V_2 (Potassium dichromate)

Assay of copper sulphate

- Dissolve the given sample in 100 mL volumetric flask by adding distilled water.
- Pipette out 20 mL of the solution in a clean Iodine flask, add 20 mL of water, 5 mL of dilute acetic acid and 10 mL of 15% KI solution.
- Keep the reaction mixture in a dark place for 5min.
- Titrate the liberated iodine against standard $Na_2S_2O_3$ until the appearance of straw yellow color.
- Add 1 mL of starch mucilage and continue the titration until the disappearance of dark blue color.
- Repeat the procedure take the average reading and calculate the amount of copper sulphate present by using the following formula.

Observation and Calculation:

Standardization of 0.1N sodium thiosulphate

Burette : 0.1 N Sodium thiosulphate
Iodine Flask : 10 mL 0.1N Potassium dichromate + 10 mL water, 2 mL of Conc. HCl + 10 mL 15% KI solution + Indicator
Indicator : Starch mucilage
End Point : disappearance of blue

CAS Vs 0.1N FAS

Sl No.	Volume of potassium dichromate (mL)	Volume of Sodium thiosulphate (0.1N)		Volume of Sodium thiosulphate consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

Calculation:

$$N_1V_1 \text{ (Sodium thiosulphate)} = N_2V_2 \text{ (potassium dichromate)}$$

$$N_1 = \frac{N_2V_2}{V_1}$$

Assay of Copper Sulphate

Burette : 0.1N Sodium thiosulphate
Iodine Flask : 20 mL Sample + 20 mL water + 5 mL dilute acetic acid + 10 mL 15% KI + 1 mL indicator
Indicator : Starch mucilage
End Point : Disappearance of dark blue color

0.1N Sodium thiosulphate Vs Sample

Sl No.	Volume of Sample (mL)	Volume of Sodium thiosulphate (0.1N)		Volume of Sodium thiosulphate consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

IP FACTOR

Each mL of 0.1N $Na_2S_2O_3$ is equivalent to 0.02497g of $CuSO_4$

Amount of sample present in 100 mL of the solution =

$$\frac{\text{Burette reading} \times \text{IP factor} \times \text{Actual normality of titrant} \times 100}{\text{IP normality} \times \text{volume of sample taken for titration}}$$

Report
The % purity of the given sample of copper sulphate was found to be _________%.

. Question for viva and synopsis

1. Explain the principle involved in the assay of Copper sulphate
2. Explain Iodometry titration.
3. How to prepare 0.1N sodium thiosulphate solution?
4. How to standardize0.1Nsodiumthiosulphatesolution??
5. Why to keep the reaction mixture in darkplacefor5min?
6. Why the starch mucilage indicator should be used towards the endpoint?
7. How to prepare starch mucilage indicator
8. Why KSCN is added to this titration?
9. Uses of Copper sulphate

Experiment No: 11

ASSAY OF CALCIUM GLUCONATE

AIM: Carry out the assay of assay of calcium gluconate by Complexometric replacement Titration.

Apparatus & Chemicals required; Burette, Pipette, Conical Flask, Volumetric Flask, 0.1N Na_2EDTA, 0.1N Magnesium sulphate, mordant black-II indicator.

Principle: Complexometric Replacement titration.

Calcium does not react with indicator satisfactory and will not give proper end point. So $MgSO_4$ is used which react with indicator and free calcium ion easily react with standard Na_2EDTA (Titrant) to produce Ca-EDTA complex. When all the Ca^{++} ion get reacted the extra amount of Na_2EDTA react with MgSO4. And at last when all the Mg^{++} form Mg-EDTA complex the extra drop of Na_2EDTA react with Indicator and show the color change.

$$Mg^{++} + Ind \rightarrow \underset{Red}{Mg\text{-}Ind}$$

$$Na_2EDTA + Ca^{++} \rightarrow Ca\text{-}Na_2EDTA + 2H^+$$

$$Mg\text{-}Ind + Na_2EDTA \rightarrow Mg\text{-}Na_2EDTA + Ind + 2H^+$$

$$Ind + Na_2EDTA \rightarrow \underset{Blue}{Ind\text{-}Na_2EDTA}$$

Procedure

Preparation & Standardization of 0.05M Na_2EDTA

- Dissolve 1.86g of Na_2EDTA in water and make up the volume 100 mL by adding distilled water which will produce approximately 0. 05M Na_2EDTA.
- 1.25 g of MgSO4 are dissolved water and make up the volume 100 mL by adding distilled water which will produce 0.05M $MgSO_4$.
- Pipette out 10 mL of 0.05M $MgSO_4$in a clean conical flask add 20 mL of distilled water and 10 mL of NH_3-NH_4Cl buffer.
- Add 2 drops of mordant black II indicator.
- Titrate the solution against prepared 0.05M Na_2EDTA until the color change from red to light blue.
- Repeat the procedure take the average reading and calculate the exact molarity of Na_2EDTA by using the formula $M_1V_{1\,(Na2EDTA)} = M_2V_{2\,(MgSO4)}$

Assay of Calcium Gluconate

- Dissolve the given sample in volumetric flask by adding distilled water.
- Pipette out 10 mL of the solution in a clean conical flask, add 20 mL of distilled water, 5 mL of 0.05M and 10 mL of NH_3-NH_4Cl buffer.
- Add 2 drops of mordant black-II indicator.
- Titrate the solution against prepared 0.05M Na_2EDTA until the color change from red to light blue.
- Repeat the procedure take the average reading and calculate the amount of sample present by using following formula.
- The volume of Na_2EDTA equivalent to $MgSO_4$ solution is subtracted from total Na_2EDTA solution used and then results are calculated.

Note; the complex formed is stable at pH 10. But each complex formed there is liberation of H^+ ion which reduce the pH of the solution. So, a basic buffer like NH_3-NH_4Cl is added to maintain the pH 10.

Observation and Calculation:

Standardization of 0.05M Na_2EDTA

Burette : 0.05 M Na_2EDTA
Conical Flask : 10 mL 0.05M $MgSO_4$ + 20 mL distilled water, 10 mL NH_3-NH_4Cl Buffer + 2 drops indicator
Indicator : Mordant black II indicator
End Point : red to light blue

0.05M Na_2EDTA Vs 0.05M $MgSO_4$

Sl No.	Volume of $MgSO_4$ (mL)	Volume of 0.05M Na_2EDTA		Volume of 0.05M Na_2EDTA consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

Calculation:

$$M_1V_{1\ (Na2EDTA)} = M_2V_{2\ (MgSO4)}$$

$$M_1 = \frac{M_2V_2}{V_1}$$

Assay of Calcium gluconate

Burette : 0.05M Na_2EDTA
Conical Flask : 10 mL Sample + 20 mL distilled water + 10 mL NH_3-NH_4Cl buffer + 2 drops indicator.
Indicator : Mordent Black-II indicator.
End Point : red to light blue color

0.05M Na_2EDTA Vs Sample

Sl No.	Volume of Sample (mL)	Volume of Na_2EDTA (0.05M)		Volume of Na_2EDTA consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

IP FACTOR
Each mL of 0.05M Na_2EDTA is equivalent to 0.02242g of Ca-gluconate

Amount of sample present in 100 mL of the solution =

$$\frac{(\text{Burette reading} - 5) \times \text{IP factor} \times \text{Actual normality of titrant}}{\text{IP normality} \times \text{volume of sample taken for titration}} \times 100$$

Report

The percentage purity of the given sample of calcium gluconate was found to be _______%.

Question for viva and synopsis

1. Explain the principle involved in the assay of Ca-gluconate.
2. How to standardize 0.05M Na_2EDTA
3. Why NH_3/NH_4Cl buffer is added in the Complexometric titration?
4. Why Calcium gluconate is titrated by Complexometric Replacement titration?

Experiment No:12

ASSAY OF HYDROGEN PEROXIDE

AIM: Carry out the assay of Hydrogen peroxide by redox Permanganometry titration.

Apparatus Required: Burette, Pipette, Conical Flask, Volumetric Flask

Chemicals Required: 0.1N $KMnO_4$ redox Parmanganometry titration, 0.1N Oxalic acid, dilute H_2SO_4.

Principle: **Redox Parmanganometry Titration**

Hydrogen peroxide is a strong reducing agent which can be easily titrated by using standard $KMnO_4$ which act as a strong oxidizing agent in presence of Sulphuric acid. There is no indicator used because $KMnO_4$ itself act as a indicator to show the end point of the titration (self-indicator) by changing the color from colorless to violet.

$$5H_2O_2+2KMnO_4+3H_2SO_4 \rightarrow K_2SO_4+2MnSO_4 +8H_2O +5O_2$$

Procedure

Preparation & Standardization of 0.1N $KMnO_4$

- Take around 0.316g of $KMnO_4$ and transfer to 50 mL beaker.
- Add little quantity of distilled water and decant to 100 mL volumetric flask.
- Repeat the process until entire $KMnO_4$ gets dissolve (no heat should be applied for dissolving $KMnO_4$).
- Finally make up the volume up to 100 mL by adding distilled water which will produce approximately 0.1N $KMnO_4$
- 0.63 g of oxalic acid dissolve in water and make up the volume 100 mL by adding distilled water which will produce 0.1N oxalic acid.
- Pipette out 10 mL of 0.1N oxalic acid in a clean conical flask add 10 mL of water and 10 mL of dilute Sulphuric acid.
- Heat the solution around 70 ^{0}C and titrate the hot solution against prepared 0.1N $KMnO_4$ until the appearance of violet color.
- Repeat the procedure take the average reading and calculate the exact normality of $KMnO_4$ by using the formula $N_1V_{1(KMnO4)} = N_2V_{2\,(Oxalic\ acid)}$

Note:

1. The solubility of $KMnO_4$ in water is low so repeated addition of water by portion is required.
2. The $KMnO_4$ is heat sensitive so we can’t apply heat to increase solubility.
3. The reactivity of oxalic acid with $KMnO_4$ is very slow. to make the reaction faster we should carry out the
   reaction at 70 ^{o}C.

Assay of Hydrogen peroxide:

- Dissolve the given sample in volumetric flask by adding distilled water.
- Pipette out 10mL of the dilute solution in a clean conical flask, add 20mL of Sulphuric acid.
- Titrate the solution against standard 0.1N $KMnO_4$ until the appearance of violet color.
- Repeat the procedure take the average reading and calculate the amount of sample present by using following formula

Observation and Calculation:

Standardization of 0.1N $KMnO_4$

Burette : 0.1 N $KMnO_4$
Conical Flask : 10 mL 0.1N Oxalic acid + 10 mL dilute H_2SO_4
Indicator : $KMnO_4$
End Point : appearance of violet color

0.1N $KMnO_4$ Vs 0.1N Oxalic acid

Sl No.	Volume of Oxalic acid (mL)	Volume of $KMnO_4$ (0.1N)		Volume of $KMnO_4$consumed (mL)	Color Change
		Initial (mL)	Final(mL)		

Calculation:

$$N_1V_{1\,(KMnO4)} = N_2V_{2\,(oxalic\ acid)}$$

$$N_1 = \frac{N_2V_2}{V_1}$$

Assay of Hydrogen peroxide

Burette : 0.1N $KMnO_4$
Conical Flask : 10 mL Sample + 20 mL sulphuric acid
Indicator : $KMnO_4$
End Point : appearance of violet color

0.1N $KMnO_4$ Vs Sample

Sl No.	Volume of Sample (mL)	Volume of $KMnO_4$(0.1N)		Volume of $KMnO_4$ consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

IP FACTOR
Each mL of 0.1N $KMnO_4$ is equivalent to 0.001701g of hydrogen peroxide

Amount of sample present in 100 mL of the solution =

$$\frac{\text{Burette reading} \times \text{IP factor} \times \text{Actual normality of titrant} \times 100}{\text{IP normality} \times \text{volume of sample taken for titration}}$$

Report
The percentage purity of the given sample of Hydrogen peroxide was found to be _______%.

Question for viva and synopsis

1. Explain the principle involved in assay of Hydrogen peroxide.
2. Explain why no indicator is used in this titration.
3. How to prepare0.1N KMnO4?
4. How to standardize0.1NKMnO4?
5. Define Redox titration
6. Why heat is applied forstandardizationof0.1NKMnO4
7. Why Heat is not applied for dissolving KMnO4.
8. How to calculate IP Factor
9. Why we should apply heat while doing the standardization.

Experiment No:13

ASSAY OF SODIUM BENZOATE

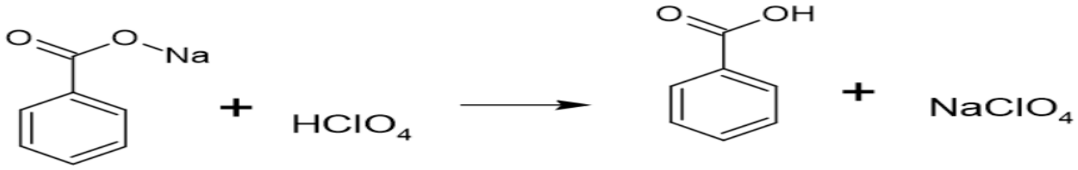
AIM: Carry out the assay of sodium benzoate by redox Parmanganometry titration

Apparatus Required: Burette, Pipette, Conical Flask, Volumetric Flask.

Chemicals Required: 0.1N Perchloric acid, glacial acetic acid, Sodium benzoate, Potassium hydrogen phthalate, Crystal Violet

Principle: **Non aqueous titration using standard Perchloric acid**

Sodium benzoate is a weakly basic substance. It is dissolve in acetic acid which exerts the leveling effect on it and increase the basic strength. Acetous Perchloric acid give rise to onium ion, which is a very strong acid and it is further increase basic strength of sodium benzoate. Now this can easily titrated with strong acid like Perchloric acid



Procedure

Preparation & Standardization of 0.1N Perchloric acid

- Perchloric acid is available as 12N strength.
- Therefore 0.1N can be prepared by diluting 120 times.
- Pipette out 8.5 mL of Perchloric acid solution, add 900 mL of glacial acetic acid with continuous stirring.
- Add 30 mL of acetic anhydride solution and make up the volume 1000 mL with glacial acetic acid, allow the solution to keep for 24 hours.

Note: Perchloric acid must be diluted with glacial acetic acid before adding acetic anhydride otherwise explosive acetyl perchlorate will be formed.

- Dissolve 2.042g of Potassium Hydrogen Phthalate in about 80 mL of glacial acetic acid.
- Heat on water bath until completely dissolves. Protect from humidity.
- Cool at 20 ^{0}C and make up the volume up to 100 mL with glacial acetic acid.
- This will produce 0.1N Potassium Hydrogen Phthalate.
- Take 10 mL of KHP solution in a clean conical flask add acetous crystal violet as indicator.
- Titrate the solution with prepare Perchloric acid until the color change from blue to green. Repeat the procedure take the average reading and calculate the exact normality of Perchloric acid by using the formula

$$N_1V_{1\ (\text{perchloric acid})} = N_2V_{2\ (\text{potassium hydrogen phthalate})}$$

Assay of Sodium Benzoate:

- Dissolve the given sample in 10 mL glacial acetic acid by warming 50 °C if necessary.
- Cool the solution at room temperature and titrate with 0.1N Perchloric acid using alpha naphthol benzoin as an indicator until the color change from blue to green.
- Repeat the procedure take the average reading. Run a blank titration and calculate the amount of sample present by using formula given below.

Observation and Calculation:

Standardization of 0.1N Perchloric acid

Burette : 0.1 N Perchloric acid
Conical Flask : 10 mL 0.1N Potassium Hydrogen Phthalate + 2 drops Indicator
Indicator : Crystal violet
End Point : Blue to green

Tabulation: Perchloric acid Vs 0.1N Potassium hydrogen phthalate

Sl No.	Volume of potassium hydrogen phthalate (mL)	Volume of Perchloric acid (0.1N)		Volume of Perchloric acid consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

Calculation:

$$N_1V_1 \text{ (Perchloric acid)} = N_2V_2 \text{ (Potassium Hydrogen phthalate)}$$

$$N_1 = \frac{N_2V_2}{V_1}$$

Blank

Burette : Perchloric acid
Conical Flask : 10 mL glacial acetic acid.
Indicator : Alpha naphthol benzein
End Point : blue to green

Blank Vs 0.1N perchloric acid

Sl No.	Volume of glacial acetic acid (mL)	Volume of perchloric acid (0.1N)		Volume of perchloric acid consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

Assay of Sodium Benzoate

Burette : Perchloric acid
Conical Flask : 10 mL Sample dissolved in glacial acetic acid.
Indicator : Alpha naphthol benzein
End Point : blue to green

Sample Vs 0.1N perchloric acid

Sl No.	Volume of Sample (mL)	Volume of perchloric acid (0.1N)		Volume of perchloric acid consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

IP FACTOR
Each mL of 0.1N Perchloric acid is equivalent to 0.01441g of sodium benzoate.

Amount of sample present in 100 mL of the solution =

$$\frac{\text{(Sample- blank) Burette reading X IP factor X Actual normality of titrant X 100}}{\text{IP normality X volume of sample taken for titration}}$$

Report:
The percentage purity of the given sample of sodium benzoate was found to be______%.

Question for viva and synopsis

1. Define non aqueous titration.
2. Explain the types of solvent used in non-aqueous titration.
3. What is onium ion?
4. What is leveling effect?
5. Explain the principle involved in the assay of sodium benzoate.

Experiment No: 14

ASSAY OF SODIUM CHLORIDE

AIM: Carry out the assay of sodium chloride by modified Volhard method

Reference: Indian Pharmacopoeia vol II, 2007, pg: 100.

Apparatus Required: Burette, Pipette, Conical Flask, Volumetric Flask

Chemicals Required:0.1N silver nitrate, 0.1N Ammonium thiocyanate, sodium chloride, Ferric ammonium sulphate and potassium dichromate

Principle: **Modified Volhard method of precipitation titration**

The sodium chloride is assayed by modified Volhard method of precipitation titration. The excess amount of standard 0.1N silver nitrated is reacted with NaCl in presence of nitric acid to form silver chloride precipitate. The extra unreacted silver nitrate is titrated against standard 0.1N ammonium thiocyanate using ferric ammonium sulphate as indicator until the appearance reddish brown color precipitate of ferric Ferro thiocyanate complex.

$$NaCl + Ag\,NO_3 \xrightarrow{\text{Dil. } HNO_3} AgCl + NaNO_3$$

$$\mathbf{Ag\,NO_3 + NH_4SCN \longrightarrow Ag\,SCN + NH_4NO_3}$$

$$Fe^{+++} + NH_4SCN \longrightarrow \underset{\text{Reddishbrown}}{Fe[Fe(SCN)_6]}$$

Procedure

Preparation and standardization 0.1N Silver Nitrate

- Dry the silver nitrate around 120 ^{0}C for 2 hours and allow to cool in a desiccator.
- Weigh 1.6987g of the dry silver nitrate.
- Dissolve in distilled and make up the volume up to 100 mL by adding distilled water. This will produce approximately 0.1N silver nitrate.
- Weigh 0.5844g of pure NaCl and dissolve in water. Make up the volume by adding distilled water up to 100 mL. This will produce 0.1N NaCl
- Pipette out 10 mL of 0.1N NaCl in a clean conical flask add 10 mL of distilled water and add 2 drops of potassium chromate solution as indicator.
- Titrate the solution against prepared 0.1N Silver nitrate until the appearance of reddish brown ppt.
- Repeat the procedure take the average reading and calculate the exact normality of silver nitrate by using the formula $N_1V_1 = N_2V_2$

Preparation & Standardization of 0.1N Ammonium thiocyanate

- Weigh accurately 0.76g of ammonium thiocyanate and dissolve in distilled. Make up the volume up to 100 mL by adding distilled water.
- This will produce approximately 0.1N ammonium thiocyanate. Pipette out 10 mL of 0.1N silver nitrate solution in a clean conical flask add 6 mL of concentrated nitric acid and 1 mL of ferric ammonium sulphate as indicator.
- Titrate the solution against prepared 0.1N ammonium thiocyanate until the appearance of reddish brown ppt.
- Repeat the procedure take the average reading and calculate the exact normality of ammonium thiocyanate by using the formula $N_1V_1 = N_2V_2$

Assay of Sodium Chloride

- Dissolve the given sample in volumetric flask by adding distilled water.
- Pipette out 25 mL of the diluted solution in a clean conical flask containing 5 mL concentrated nitric acid,
- Add 10 mL of 0.1N $AgNO_3$ and 2-3 mL of nitrobenzene, 1 mL of ferric ammonium sulphate indicator.
- Shake vigorous to coagulate the ppt.
- Titrate the residual silver nitrate with standard 0.1N ammonium thiocyanate until the appearance reddish brown color.
- Repeat the procedure take the average reading and calculate the amount of sample present by using following formula.
- From the volume of silver nitrate solution added, subtract the volume of silver nitrate solution that is equivalent to the volume of standard ammonium thiocyanate required.

Observation and Calculation:

Standardization of 0.1N Silver Nitrate

Burette : 0.1 N Silver Nitrate
Conical Flask : 10 mL 0.1N NaCl + 10 mL distilled water + 2 drops Indicator
Indicator : Potassium Chromate solution
End Point : Appearance of reddish brown precipitate

Silver Nitrate Vs 0.1N Potassium hydrogen phthalate

Sl No.	Volume of NaCl (mL)	Volume of Silver Nitrate (0.1N)		Volume of Silver Nitrate consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

Calculation:

$$N_1V_{1\ (\text{Silver Nitrate})} = N_2V_{2\ (\text{NaCl})}$$

$$N_1 = \frac{N_2V_2}{V_1}$$

Standardization of 0.1N Ammonium thiocyanate

Burette : 0.1N Ammonium thiocyanate
Conical Flask : 10 mL 0.1N Silver Nitrate + 6 mL Concentrated HNO_3 + 1 mL Indicator
Indicator : Ferric ammonium sulphate solution
End Point : appearance of reddish brown color

0.1N Ammonium thiocyanate Vs 0.1N Silver Nitrate

Sl No.	Volume of Silver Nitrate (mL)	Volume of Ammonium thiocyanate (0.1N)		Volume of Ammonium thiocyanate consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

Calculation:

$$N_1V_1 \text{ (Ammonium thiocyanate)} = N_2V_2 \text{ (Silver nitrate)}$$

$$N_1 = \frac{N_2V_2}{V_1}$$

Assay of Sodium Chloride

Burette : 0.1N Ammonium thiocyanate
Conical Flask : 10 mL 0.1N $AgNO_3$ + 2-3 mL nitrobenzene + 1 mL indicator
Indicator : ferric ammonium sulphate solution
End Point : Reddish brown colour

Sample Vs 0.1N Ammonium thiocyanate

Sl No.	Volume of Sample (mL)	Volume of Ammonium thiocyanate (0.1N)		Volume of Ammonium thiocyanate consumed (mL)	Color Change
		Initial (mL)	Final (mL)		

IP FACTOR
Each mL of 0.1N silver nitrate is equivalent to 0.0005849g of NaCl

Amount of sample present in 100 mL of the solution =

$$\frac{(N \times 50)_{AgNO_3} - (N \times \text{ Burette reading})_{\text{ammonium thiocyanate}} \times \text{IP factor} \times \text{Actual normality of titrant} \times 100}{\text{IP normality} \times \text{volume of sample taken for titration}}$$

Report

The percentage purity of the given sample of sodium chloride was found to be_____%.

Question for viva and synopsis

1. Explain the principle involved in the assay of NaCl.
2. Why Nitro benzene used in the assay of NaCl.
3. Explain Volhard and Mohr's method of Precipitation titration

Experiment No: 15

PREPARATION OF BORIC ACID

AIM: To prepare and submit pure product of boric acid from borax and calculate the % yield.

Apparatus Required: Beaker 100 mL, glass rod, funnel, filter paper.

Chemicals Required: borax, dil.Sulphuric acid, distilled water

Principle:
Boric acid is an odorless white crystalline powder or colorless shiny plates unctuous to the touch or white crystals. It is soluble in hot water and boiled ethanol and glycerin. It is not meant for internal use.

Uses:
1. It is used as local anti-infective.
2. 5% solution of boric acid is used to maintain isotonic solution.
3. It is used to prepare buffer solution.
4. It is used to prepare different cosmetic preparation.

Preparation:
- Add 1.2 mL of concentrated Sulphuric acid to 6 mL of water and heat the solution.
- In another beaker dissolve 6 g of borax in 16 mL of water.
- Add hot solution of Sulphuric acid to the hot solution of borax with constant stirring.
- The hot solution is now filter and keep aside for crystallization.
- The boric acid crystals are filtered; washed with cold water and dried at room temperature. Calculate the % yield of boric acid.

Reaction:

$$Na_2B_4O_7 + H_2SO_4 + 5\ H_2O \rightarrow Na_2SO_4 + 4H_3BO_4$$

Calculation:

Mol wt of borax = 381.24

Mol wt of Boric acid = 61.83

Theoretical yield for 6g borax = $\frac{6 \times (4 \times 61.83)}{381.24}$

= 3.891 g

% yield = $\frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100$

Report:
The percentage yield of the prepared boric acid was found to be____________%.

Experiment No: 16

PREPARATION OF MAGNESIUM SULPHATE

AIM: To prepare and submit pure product of magnesium sulphate and calculate the % yield

Apparatus Required: Beaker 100 mL, glass rod, funnel, filter paper, distilled water

Chemicals Required: $MgCO_3$, Sulphuric acid.

Discussion:
It is white crystalline odorless powder. It is soluble in water but freely soluble in hot water and partially soluble in ethanol.

Uses:
It is used as osmotic laxative and used in the treatment of electrolyte deficiency.

Preparation:

- Warm 20 mL of dilute Sulphuric acid in a beaker.
- Add 4 g of $MgCO_3$ in portions until it stops reacting (no more fizzing) and there is some solid clearly visible in the mixture.
- Keep the mixture at room temperature for 15 min, filter it.
- Evaporate the filtrate to half its original volume.
- Allow to stand and crystallize. Wash the crystals with distilled water.

Calculation:

Mol wt of $MgCO_3$ = 84.38g
Mol wt of $MgSO_4$ = 120.38g

$$\% \text{ yield} = \frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100$$

Report:
The percentage yield of the prepared magnesium sulphate was _______%

Experiment No:17

PREPARATION OF ZINC OXIDE

AIM: To prepare and submit pure product of zinc oxide and calculate the % yield

Apparatus Required: Beaker 100 mL, glass rod, funnel, filter paper

Chemicals Required: Distilled water, $ZnSO_{4,}$ $NaCO_3$

Discussion:
It occurs as a soft, white or faintly white, very fine powder, free from grittiness. It is odorless and tasteless. When exposed to air it slowly absorbs carbon dioxide from air. It is insoluble in water and alcohol.

Uses: mild antiseptic and an astringent.
In the form of ointment or dusting powder, it is used in the treatment of skin diseases.
It is employed in the treatment of eczema, ringworm, psoriasis.

Preparation:

- Dissolve 2 g of Zinc sulphate in 10 mL water in a beaker.
- Dissolve 2 g Sodium carbonate in 10 mL water and boil it.
- Add the solution of Zinc sulphate to boiling Sodium carbonate solution.
- The precipitate of Zinc carbonate is collected, washed until it becomes free from sulphate.
- Dry and ignite to get zinc oxide.

Calculation:

Mol wt of $ZnSO_4$ =
Mol wt of ZnO =
% yield = $\frac{\text{Practical yield}}{\text{Theoretical yield}}$ X 100

Report:
The percentage yield of prepared zinc chloride was found to be __________%.

Experiment No:18

PREPARATION OF CALCIUM CARBONATE

AIM: To prepare and submit pure product of calcium carbonate and calculate the % yield

Apparatus Required: Beaker 100 mL, glass rod, funnel, filter paper

Chemicals Required: Distilled water, sodium carbonate, calcium chloride.

Discussion:
It occurs as fine white, micro-crystalline powder. It is odorless and tasteless.
It is unstable is water and alcohol.

Uses: Externally used as a dentifrice because of its abrasive property.
Internally it is used as an antacid, since it produces alkalosis continued use as an antacid is not advisable.

Preparation:

- Dissolve 2 g of Calcium Chloride in 10 mL water in a beaker.
- Dissolve 2 g Sodium carbonate in 10 mL water.
- Boil both the solutions and mix both while hot.
- Filter the precipitate, Dry and calculate the percentage purity.

Calculation:

Mol wt of $CaCl_2$ =
Mol wt of $CaCO_3$ =
% yield = $\frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100$

Report:

The percentage yield of the prepared calcium carbonate was found to be___________%

Experiment No:19

PREPARATION OF ALUMINIUM HYDROXIDE

AIM: To prepare and submit pure product of aluminum hydroxide and calculate the % yield

Apparatus Required: Beaker 100 mL, glass rod, funnel, filter paper

Chemicals Required: Distilled water, potash alum, sodium carbonate.

Discussion:
It occurs as aqueous white viscous suspension of hydrated aluminum oxide having varying amounts of basic aluminum carbonate.

Uses:
Aluminum hydroxide gel is a very effective slow-acting antacid.
It is widely used in the treatment of hyperchlorydria.

Preparation:
Dissolve 5.16 g of potash alum in 10 mL water in a beaker.
Dissolve 3.15 g Sodium carbonate in 10 mL water and boil it.
Slowly add the solution of potash alum to boiling Sodium carbonate solution.
The precipitate of Aluminum hydroxide is collected, washed until it becomes free from sulphate.

Calculation:

Mol wt of $2KAl\,(SO_4)_2$ =
Mol wt of $Al\,(OH)_3$ =
% yield = $\frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100$

Report:
The percentage yiled of the prepared Aluminium Hydroxide was found to be______%.

Experiment No:20

PREPARATION OF FERROUS SULPHATE

AIM: To prepare and submit pure product of Ferrous Sulphate and calculate the % yield

Apparatus Required: Beaker 100 mL, glass rod, funnel, filter paper, distilled water

Chemicals Required: Iron Fillings, Sulphuric acid.

Principle:
Ferrous sulphate contain not less than 98% and not more than 105% of $FeSO_4 .7H_2O$.The principle behind formation of ferrous sulphate is simple displacement reaction, with evolution of hydrogen gas. Ferrous Sulphate is prepared by reaction between iron fillings and dilute sulphuric acid.

Uses:
It is used in the treatment of anemia caused by iron deficiency.

Preparation:

- Take 20 ml of dil.sulphuric acid in a beaker.Add 2 gm of iron fillings in a beaker with gradual heating until effervescences ceases.
- After completion of reaction the liquid is concentrated by boiling. Then the solution is filtered and allowed to cool.
- The crystals are separated and dried, then recrystallise from water.

Calculation:

Mol wt of Fe = 56 g
Mol wt of $FeSO_4$ = 152g

$$\% \text{ yield} = \frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100$$

Report:
The percentage yield of the prepared Ferrous sulphate was___________%

Experiment No:21

PREPARATION OF POTASH ALUM

AIM: To prepare and submit pure product of Potash Alum and calculate the % yield

Apparatus Required: Beaker 100 mL, glass rod, funnel, filter paper, distilled water.

Chemicals Required: Potassium sulphate, aluminum sulphate.

Principle:
Potash alum is prepared by dissolving an equimolar mixture of hydrated aluminum sulphate and potassium sulphate in minimum amount of water containing a little sulphuric acid and then subjecting the resulting solution to the crystallization, when octa hydral crystals of potash alum is separate out.

Uses: It is used for water purification and also as antiseptics.
It can be used as astringent.
It can be used as deodorant.

Preparation:

- Take 250 ml beaker. Wash it with water and then transfer 2.5 g potassium sulphate crystals to it. Add about 20 ml of water .Stir to dissolve the crystals. Warm it if required.
- Take other 250 ml beaker, wash it with water and then transfer 10 gm aluminum sulphate crystal to it. Add about 20 ml of water and 1 ml of dil.sulphuric acid Heat for 5 mins.
- Mix both the solution in a china dish and place the china dish on wire gauze placed over a burner
- Concentrate the solution. Soon the crystals of Potash alum are separate out.
- Filter the residue and dry it in air.

Reaction:

$$Al_2(S0_4) + k_2SO_4 + H_2O \longrightarrow KAl(SO_4)_3.12\,H_2O$$

Calculation:

Mol wt of $Al_2(SO_4)$ = 342 g
Mol wt of Potash alum = 474 g
% yield = $\frac{\text{Practical yield}}{\text{Theoretical yield}}$ X 100

Report:
The percentage yield of prepared zinc chloride was

Experiment No:22

SWELLING POWER OF BENTONITE

AIM: To Study swelling power of Bentonite

Apparatus Required: Graduated measuring cylinder.

Chemicals Required: Bentonite, Sodium lauryl sulphate.

Principle:
Bentonite is natural colloidal, hydrated aluminum silicate that has been processed to remove grit and non-swelling component of ore.It is under the category of Pharmaceutical aid. Bentonite is insoluble and does not swell in organic solvents.

Uses: It can be used as bulk laxative.
It is Pharmaceutical aid used as suspending agent.
It can be used a desiccant due to its adsorption property.

Procedure: Add 2 gm of bentonite in twenty portions at intervals of 2 minutes to 100 ml of a 1% w/v solution of sodium lauryl sulphate in a 100 ml graduated cylinder about 3 cm In diameter. Allow each portion to settle before adding the next and let it stand for 2 Hours.

Conclusion: The apparent volume of the sediment at the bottom of the cylinder has to be 24 ml or more, and then sample passes the test for swelling power of bentonite.

Experiment No:23

IDENTIFICATION OF CATIONS

AIM: Identify the following Cations ions, Sodium, potassium, Calcium, Iron

Identification for Na^+

Test	Observation	Inference
FLAME TEST Sample moistened with HCl, introduce on platinum wire and burn on flame	Yellow color flame	Na^+ ion present
Sample + potassium antimonite solution. Boil and scratch the side of the test tube with glass rod	Dense white ppt; of disodium antimonite	Na^+ ion present

$$Na^+ + K_2H_2SbO_4 \rightarrow Na_2H_2SbO_4 + 2K^+$$

Test	Observation	Inference
To the sample solution add dilute acetic acid and magnesium urinyl acetate	Yellow crystal	Na^+ ion present

$$3Na^+ + 3UO_2(CH_3COO)_2 + Mg(CH_3COO)_2 + CH_3COOH + 4H_2O \rightarrow 3[NaMgUO_2(CH_3COO)_2]\,4H_2O$$

Identification for K^+

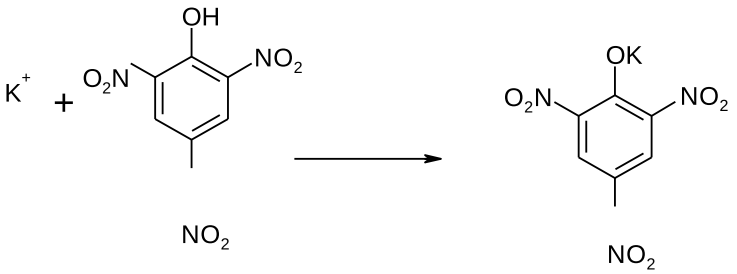
Test	Observation	Inference
To the sample solution add freshly prepare Sodium cobalt nitrate solution	Yellow ppt	K^+ ion present

$$3K^+ + Na_3[CO(NO_2)_6] \rightarrow 3Na^+ + K_3[CO(NO_2)_6$$

Test	Observation	Inference
To the sample solution add concentrated solution of tartaric acid in alcohol	White ppt	K^+ ion present

$$K^+ + C_4H_6O_6 \rightarrow H^+ + C_4H_5O_6K$$

Test	Observation	Inference
Sample + picric acid	Yellow ppt	K^+ ion present



Identification for Ca^{++}

Test	Observation	Inference
Sample solution in HCl. Neutralize with NaOH and treated with ammonium carbonate	white ppt	Ca^{++} ion present

$$Ca^{++} + (NH_4)_2CO_3 \rightarrow CaCO_3 + NH_4^+$$

Test	Observation	Inference
Sample solution + ammonium oxalate	white ppt of Ca-oxalate is obtain which is soluble in HCl	Ca^{++} ion present

$$Ca^{++} + (NH_4)_2(COO)_2 \rightarrow Ca\,(COO)_2 + 2\,NH_4^+$$

Test	Observation	Inference
Concentrated Sample solution + potassium chromate solution and shake well	Yellow crystal of Ca-chromate which dissolve in water	Ca^{++} ion present

$$Ca^{++} + K_2CrO_4 \rightarrow K^+ + CaCrO_4$$

Identification for Fe^{++}

Test	Observation	Inference
Sample solution + Potassium ferrocyanide	White ppt which convert to blue on air oxidation	Fe^{++} ion present

$$Fe^{++} + 2KFe(CN)_6 \rightarrow K_2Fe[Fe(CN)_6]_2$$

$$4K_2Fe\,[Fe(CN)_6]_2 + 4H^+ + O_2 \rightarrow 4KFe[Fe(CN)_6] + 4K + 2H_2O$$

Test	Observation	Inference
Aqueous solution of Sample + Potassium ferricyanide solution	Dark blue ppt which insoluble in dil HCl but decompose by NaOH solution	Fe^{++} ion present

Experiment No:24

IDENTIFICATION OF ANIONS

AIM: To Identify the following Ions, Bicarbonates, Chlorides, Sulphates and Iodides

Identification for HCO_3^-

Test	Observation	Inference
Sample + HCl or H_2SO_4	Effervescence observed	HCO_3^- ion present

$$HCO_3^- + HCl \rightarrow H_2O + CO_2 + Cl^-$$

Test	Observation	Inference
Sample + $MgSO_4$ and boil	White ppt	HCO_3^- ion present

$$HCO_3^- + MgSO_4 \rightarrow Mg(HCO_3)_2 \rightarrow MgCO_3 + H_2O + CO_2$$

Test	Observation	Inference
Sample + $HgCl_2$	White ppt	HCO_3^- ion present

$$HgCl_2 + HCO_3^- \rightarrow Hg(HCO_3)_2 + 2Cl^-$$

Identification for Cl^-

Test	Observation	Inference
Sample + Dilute HNO_3 + Ag NO_3	White ppt	Cl^- ion present

$$Cl^- + AgNO_3 \xrightarrow{\text{Dil } HNO_3} AgCl + NO_3^-$$

Test	Observation	Inference
Take solid sample in a dry test tube. Add potassium dichromate and Sulphuric acid. Transferred the red color chromoyl chloride to another test tube containing water and lead acetate	Yellow ppt	Cl^- ion present

$$K_2Cr_2O_7 + 2\,Cl^- + 6\,H_2SO_4 \rightarrow 6\,HSO_4^- + 3\,H_2O + Cr_2O_2Cl_2$$

$$Cr_2O_2Cl_2 + H_2O \rightarrow H_2CrO_4 + HCl$$

Identification for $SO_4^{=}$

Test	Observation	Inference
Aqueous solution of sample; add barium chloride. Acidified with HCl	White ppt	$SO_4^{=}$ion present

$$SO_4^{=} + BaCl_2 \xrightarrow{\text{Dil HCl}} BaSO_4 + 2Cl^-$$

Test	Observation	Inference
Aqueous solution of sample; add lead acetate solution	White ppt of lead sulphate is formed which is soluble in ammonium acetate and NaOH solution	$SO_4^{=}$ion present

$$SO_4^{=} + Pb\,(CH3COO)_2 \rightarrow PbSO_4 + 2\,CH_3COO^-$$

Identification for I^-

Test	Observation	Inference
Aqueous solution of sample acidified with Sulphuric acid and potassium dichromate. Add chloroform and shake vigorously	Chloroform layer become violet	**I^-** ion present

$$6\,I^- + K_2Cr_2O_7 + 7H_2SO_4 \rightarrow 3\,I_2 + 7H_2O + K_2SO_4 + 2\,CrSO_4^{3-}$$

Test	Observation	Inference
Sample solution + KIO_3 and dilute acetic acid + 1 mL of starch mucilage	Blue colour produce	I^- ion present

$$5\,I^- + 6\,CH_3COOH + KIO_3 \rightarrow 3I_2 + 6\,CH3COO^- + 3H_2O$$

$$I_2 + \text{starch} \rightarrow \text{blue color complex}$$

Test	Observation	Inference
Sample + Dilute HNO_3 + Ag NO_3	Pale tallow ppt of silver iodide	I^- ion present

$$I^- + AgNO_3 \xrightarrow{\text{dil } HNO_3} AgI + NO_3^-$$

Test	Observation	Inference
Aqueous solution of sample + $HgCl_2$	Pale tallow ppt of silver iodide	I^- ion present

$$2I^- \ + \ HgCl_2 \rightarrow 2Cl^- \ + \ \underset{\text{Red}}{HgCl_2}$$

$$HgCl_2 \ + \ 2KI \ \rightarrow HgI_4^-$$

Question for viva and synopsis

1. Write the identification test with reaction of Na^+, K^+, Ca^{++}, $Fe^{++,}$ HCO_3^-, Cl^-, SO_4^-, I^-

Vision and Mission of the Institution

Vision

The East Point College of Pharmacy aspires to be a globally acclaimed institution, **recognized** for **excellence in** pharmaceutical education, research and nurturing students for **holistic development.**

Mission

M1 Create pharmacy graduates through **quality education**

M2 Promote innovation, **creativity**, and excellence **in teaching**, learning, and **research**

M3 **Inspire** integrity, teamwork, critical thinking**, personal** development, and ethics in **students** and lay **the** foundation for lifelong learning

M4 **Serve** the **healthcare, technological, scientific**, and **economic** needs of then **society**.