SUBJECT: PHARMACEUTICAL INORGANIC CHEMISTRY

PRACTICAL MANUAL BOOK

LAB MANUAL
PHARMACEUTICAL INORGANIC CHEMISTRY

(B. Pharm 1ST SEM)

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CONTENTS

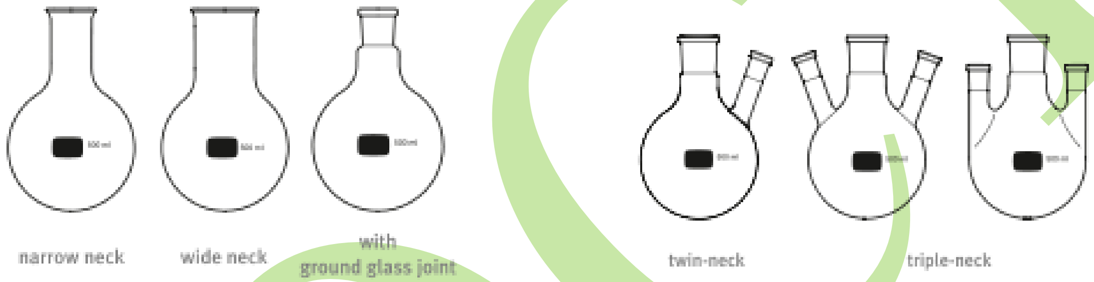
EXPERIMENT - 1

INTRODUCTION TO GLASSWARE & EQUIPMENTS

In chemistry a variety of glassware and techniques are used for the preparation, separation and purification of organic compounds.

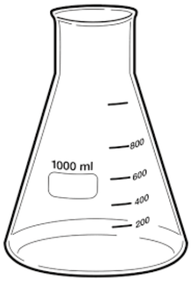
1) **FLASKS:** These are the common type of glassware used for refluxing & distillation. The different types of flasks are

   1) Round bottomed flask

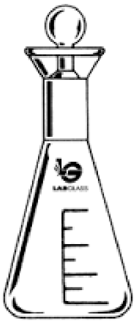


   2) Volumetric flask (measuring flask or graduated flask)

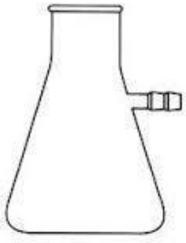
   3) Conical flask (Erlenmeyer flask)



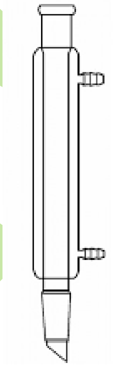
4) Iodine flask (stoppered flask)



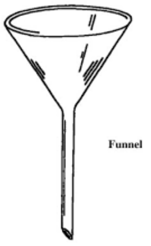
5) Buchner’s flask



2) **CONDENSERS:** Air condenser and water condenser are used for refluxing & also ordinary distillation. The air condenser is employed if the liquid has very high boiling point.



3) **FUNNELS:** Three types of funnels are generally used 1) Ordinary 2) Buchner and 3) Separatory funnel.



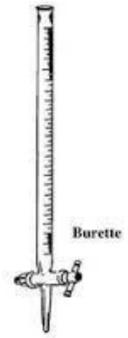
4) **ADAPTORS:** These are normally used for delivery of distillate from the condenser to the receiver vacuum also can be applied to the adaptor if required.

5) **PIPETTE:** It is a glass tube which indicates at the centre to bear the volume of liquid as marked on it may be cylindrical with graduation .They may be of 1ml, 2ml, 5ml, 10ml, 25ml etc.

Bulb Pipette

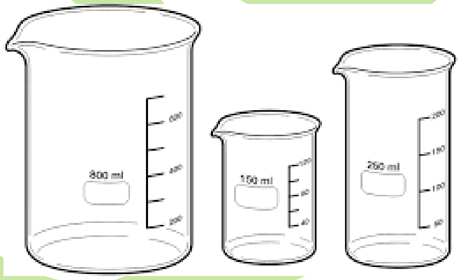
Graduated pipette

6) **BURETTE:** It is a long graduated tube with stop cork at one end. It is made up of glass or polyvinylchloride in different volumes. It can be used to transfer or measure a desired volume of liquid. Each ml of the volume of liquid can be read on the graduated surface of the burette.



7) **MEASURING CYLINDER:** It is a tall cylinder made up of thick glass and is graduated. It is available in the capacity of 5ml, 10ml, 25ml, 50ml, 100ml, etc. It is used to measure a definite volume of a liquid.

8) **BEAKERS:** These are cylindrical, flat bottomed containers available in the capacities of 25ml, 50ml,100ml etc the volume of the beaker is noted on the face of the beaker.



EQUIPMENTS:

1) **DESSICATOR:** It is a covered glass container designed for the storage of compounds in a dry atmosphere. It usually contains a drying agent in the bottom part separated by means of porcelain plate having holes in it through the surface. The drying agents generally used are silica gel, calcium chloride, activated aluminum. It should be noted that a dessicator whose vapor pressure is greater than that of substance itself. These are of two types: ordinary and vacuum dessicator.

2) **STIRRERS:** These are generally made of glass but those made of stainless steel or Teflon is also used. The stirrer is attached to a small electric motor with the aid of small pressure tubing & mechanical agitation is achieved.

   1) Magnetic stirrer: It is used for stirring small quantities of non-viscous reaction mixtures. The stirring is achieved by a magnetic spinning bar which is added to the reaction mixture.

2) Mechanical stirrer: Large scale mixtures &viscous reaction mixtures require great power for uniform stirring this is achieved by an electric motor attached to a stirrer blade.

GOOD LABORATORY PRACTICES

1. Wear neat and ironed laboratory apron while working
2. Come well prepared by reading the principle and procedure for the experiment concerned.
3. Read the procedure and/or consult lab in-charges for solving your problem or clearing your doubts.
4. Do not consult your classmates for doubts which often do not give the benefit.
5. Maintain discipline and norms
6. Use clean glass wares.
7. Use strong acids, alkalies and other corrosives carefully.
8. Do not displace the reagents from their respective places.
9. Do not interchange pipettes/ droppers from one reagent to the other without thorough cleaning.
10. Use the gas whenever necessary; close the knob when not required.
11. Prepare your own reagents for correct results.
12. Submit laboratory record for correction in every practical class
13. Take signature from your lecturer for all the observations.
14. Leave the laboratory only after cleaning your work bench.

EXPERIMENT - 2

LIMIT TEST FOR CHLORIDES

AIM: To perform the limit test for chlorides in the given sample**.**

APPARATUS REQUIRED: Nessler's cylinders, glass rod, beaker and pipette.

CHEMICALS REQUIRED: Standard Sodium Chloride solution, dilute Nitric acid, 0.1 M Silver natrate solution and Hydrochloric acid.

PRINCIPLE: This test is designed for the control of chloride impurity in inorganic substances. It depends upon the precipitation of chlorides upon reaction with silver nitrate in the presence of nitric acid. The extent of precipitation depends upon the amount of silver chloride formed i.e. on the amount of chloride ions present in the substance. The opalescence produced was compared with a reference standard opalescence having a known amount of chloride impurity.

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

PROCEDURE:

Take two Nessler's Cylinders, wash the nd then label as **STANDARD** and **TEST**

Preparation of Standard chloride solution: Accurately weigh 0.05845g of NaCl and dissolve in 100 ml of distilled water. (0.05845% w/v of NaCl).

Preparation of standard Solution:

Take 1 mL of NaCl solution and transfer into Nessler's cylinder and add 10 ml dilute Nitric Acid and make up the volume up to 50 mL with distilled water then add 1 mL of silver nitrate solution. Stir well with a glass rod and keep it aside for 5 min.

Preparation of Test Solution:

Weigh a specified quantity of test sample and transfer into Nessler's cylinder and add 10 ml dilute Nitric Acid and make up the volume up to 50 mL with distilled water then add 1 mL of silver nitrate solution. Stir well with a glass rod and keep it aside for 5 min.

SUMMARY:

STANDARD SOLUTION	**Observation**	**Inference**
In a Nessler's cylinder Chloride std. solution (25ppm Cl) 10ml taken. Add 5ml water + 10 ml of dil.HNO3. Dilute to 50 ml in Nessler's cylinder + 1 ml of 0.1 M $AgNO_3$ solution. Stir & allow to stand for 5 min. Observe the opalescence	The opalescence produced in sample solution is **greater than** / **less than** that of standard solution.	The sample **passes/ doesn't pass** the limit test for chlorides.
TEST SOLUTION Specified substance (1gm) + 10 ml of water + 10 ml of dil.HNO_3. Dilute to 50 ml in Nessler's cylinder + 1 ml of 0.1 M $AgNO_3$ solution. Stir & allow to stand for 5 min. Observe the opalescence		

The opalescence produced in the test solution is compared with that produced in standard solution.

OBSERVATION:

REPORT: The sample________limit test for chlorides.

VIVA QUESTIONS:

1. What is impurity.
2. Define limit test.
3. What type of apparatus is used for performing limit test?
4. Write the principle involved in the limit test for chlorides.
5. How is standard solution prepared?
6. What type of reagent used in limit test for chlorides?
7. On what criteria the standard and test solutions are compared?
8. What is the purpose of dilute nitric acid?
9. What forms the precipitate in limit test for chlorides?
10. Define opalescence.
11. What is turbidity?

EXPERIMENT - 3

LIMIT TEST FOR SULPHATES

AIM: To perform limit test for sulphates in the given sample.

APPARATUS REQIURED: Nessler's cylinder, beaker, pipette, measuring cylinder, glass rods.

CHEMICALS REQUIRED: $BaCl_2$ solution, $BaSO_4$ reagent, dil. HCl, standard solution of potassium sulphate, distilled water, test sample,

PRINCIPLE: This test is designed for the control of sulphate impurity in inorganic substances. It depends upon the precipitation of the sulphate with barium chloride in the presence of HCl and traces of barium sulphate. The turbidity obtained is compared with standard solution, which contains the known amount of sulphate ions under the same experimental conditions.

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

PROCEDURE:

Preparation of $BaSO_4$ reagent: This reagent must be freely prepared by mixing 10 ml of 25 % solution of $BaCl_2$ and 15 ml of ethanolic sulphate standard solution, which contains 0.10891% K_2SO_4 in 3% ethanol. K_2SO_4 has been added to increase the sensitivity of the test. Very small amount of barium sulphate present in the reagent acts as seeding agent for precipitation of barium sulphate. Alcohol helps in preventing super saturation of barium sulphate.

Preparation of test solution: Dissolve a specified quantity of substance in water or prepare solution as directed by I.P and transfer to Nessler's cylinder. To this add 2 ml of dilute HCl and make the volume to 45 ml with water. And add 5 ml of $BaSO_4$ reagent. Stir and keep it aside for 5 min.

Preparation of standard solution: Take 1ml of 0.1089% w/v of K_2SO_4 in Nessler's cylinder and add 2 ml of HCl and transfer to Nessler's cylinder and make the volume to 45 ml with H_2O. To this add 5 ml of $BaSO_4$ reagent. Stir and keep aside for 5 min. Finally compare the turbidity of two solutions.

SUMMARY:

	Observation	**Inference**
STANDARD SOLUTION Take 1ml of 0.1089 % w/v solution of potassium sulphate in Nessler's cylinder + 2ml of dilute hydrochloric acid + Dilute to 45 ml in Nessler's cylinder + 5ml of barium sulphate reagent. Keep aside for 5 min.	The opalescence produced in sample solution is **greater than / less than** that of standard solution.	The sample **passes/ doesn't pass** the limit test for sulphates.
TEST SOLUTION Take specific weight(1gm) of sample + Add 2ml of dilute hydrochloric acid +Dilute to 45 ml in Nessler's cylinder + 5ml of barium sulphate reagent. Keep aside for 5 min.		

The opalescence produced in the test solution is compared with that produced in standard solution.

OBSERVATION:

REPORT: The given sample__________the limit test for sulphates.

VIVA QUESTIONS:

1. Write the principle involved in limit test for sulphates.
2. What forms precipitate in limit test for sulphates?
3. How barium sulphate reagent is prepared?
4. The standard solution is prepared with_________.
5. What is the importance of dilute hydrochloric acid in limit test for sulphates?
6. What is seeding agent?
7. What is the importance of ethanol in limit test for sulphates?
8. Write reaction involved in limit test for sulphate.

EXPERIMENT - 4

MODIFIED LIMIT TEST FOR CHLORIDES

AIM: To perform modified limit test for chlorides for given sample

APPARATUS: Nessler cylinder, pipette, glass rod, measuring cylinder.

CHEMICALS REQUIRED: NaCl, dilute nitric acid, Distilled water, Silver nitrate, Ethanol, Potassium sulphate, Barium sulphate, Potassium permanganate.

PRINCIPLE:

Potassium permanganate gives pink colour in aqueous solution which interfere with the general limit test. To decolorize potassium permanganate it is reduced by heating with ethanol, the precipitate formed is removed by filtration. The colourless filtrate obtained is subjected to limit test.

PROCEDURE:

1mg of potassium permanganate is heated with 4ml of ethanol until the colour becomes colourless. Filter the solutions for comparing with standards, use 20ml of filtrate and complete the limit test.

Take two Nessler's Cylinders, wash them thoroughly and then label as **STANDARD** and **TEST**

Preparation of Standard chloride solution: Accurately weigh 0.05845g of NaCl and dissolve in 100 ml of distilled water. (0.05845% w/v of NaCl).

Preparation of standard Solution:

Take 1 ml of NaCl solution and transfer into Nessler's cylinder and add 10 ml dilute Nitric Acid and make up the volume up to 50 ml with distilled water then add 1 ml of silver nitrate solution. Stir well with a glass rod and keep it aside for 5 min.

Preparation of Test Solution:

Take 20ml of above prepared sample and transfer into Nessler's cylinder and add 10 ml dilute Nitric Acid and make up the volume up to 50 ml with distilled water then add 1 ml of silver nitrate solution. Stir well with a glass rod and keep it aside for 5 min.

SUMMARY:

	Observation	**Inference**
STANDARD SOLUTION In a Nessler's cylinder Chloride std. solution (25ppm Cl) 10ml taken. Add 5ml water + 10 ml of dil.HNO3. Dilute to 50 ml in Nessler's cylinder + 1 ml of 0.1 M $AgNO_3$ solution. Stir & allow to stand for 5 min. Observe the opalescence	The opalescence produced in sample solution is **greater than / less than** that of standard solution.	The sample **passes/ doesn't pass** the limit test for chlorides.
TEST SOLUTION 20ml of test solution + 10 ml of dil.HNO_3. Dilute to 50 ml in Nessler's cylinder + 1 ml of 0.1 M $AgNO_3$ solution. Stir & allow to stand for 5 min. Observe the opalescence		

The opalescence produced in the test solution is compared with that produced in standard solution.

OBSERVATION:

REPORT: The sample_________limit test for chlorides.

Viva Questions

1. What is the use of potassium permanganate.

EXPERIMENT - 5

MODIFIED LIMIT TEST FOR SULPHATES

AIM: To perform the modified limit test for sulphates for given sample

APPARATUS: Nessler cylinder, pipette, glass rod, measuring cylinder.

CHEMICALS REQUIRED: NaCl, dilute nitric acid, Distilled water, Silver nitrate, Ethanol, Potassium sulphate, Barium sulphate, Potassium Permanganate.

PRINCIPLE:

Potassium permanganate gives pink colour in aqueous solution which interfere with the general limit test. To decolorize potassium permanganate it is reduced by heating with ethanol, the precipitate formed is removed by filtration. The colourless filtrate obtained is subjected to limit test.

PROCEDURE:

1mg of potassium permanganate is heated with 4ml of ethanol until the colour becomes colourless. Filter the solutions for comparing with standards, use 20ml of filtrate and complete the limit test.

Preparation of $BaSO_4$ reagent: This reagent must be freely prepared by mixing 10 ml of 25 % solution of $BaCl_2$ and 15 ml of ethanolic sulphate standard solution, which contains 0.10891% K_2SO_4 in 3% ethanol. K_2SO_4 has been added to increase the sensitivity of the test. Very small amount of barium sulphate present in the reagent acts as seeding agent for precipitation of barium sulphate. Alcohol helps in preventing super saturation of barium sulphate.

Preparation of test solution: Take 20ml of above decolourised sample and transfer into Nessler's cylinder. To this add 2 ml of dilute HCl and make the volume to 45 ml with water. And add 5 ml of $BaSO_4$ reagent. Stir and keep it aside for 5 min.

Preparation of standard solution: Take 1ml of 0.1089% w/v of K_2SO_4 in Nessler's cylinder and add 2 ml of HCl and transfer to Nessler's cylinder and make the volume to 45 ml with H_2O. To this add 5 ml of $BaSO_4$ reagent. Stir and keep aside for 5 min. Finally compare the turbidity of two solutions.

SUMMARY:

STANDARD SOLUTION	**Observation**	**Inference**
Take 1ml of 0.1089 % w/v solution of potassium sulphate in Nessler's cylinder + 2ml of dilute hydrochloric acid + Dilute to 45 ml in Nessler's cylinder + 5ml of barium sulphate reagent. Keep aside for 5 min.	The opalescence produced in sample solution is **greater than / less than** that of standard solution.	The sample **passes/ doesn't pass** the limit test for sulphates.
TEST SOLUTION		
Take 20ml of sample + Add 2ml of dilute hydrochloric acid +Dilute to 45 ml in Nessler's cylinder + 5ml of barium sulphate reagent. Keep aside for 5 min.		

The opalescence produced in the test solution is compared with that produced in standard solution.

OBSERVATION:

REPORT: The given sample_________the limit test for sulphates.

EXPERIMENT - 6

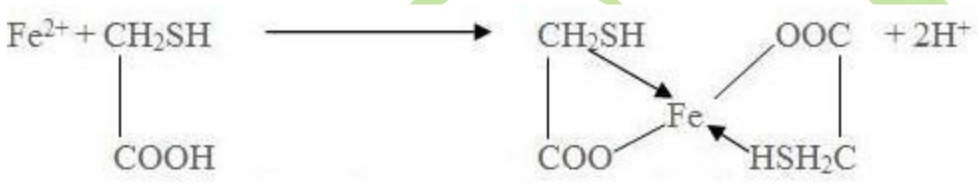
LIMIT TEST FOR IRON

AIM: To perform limit test for iron in the given sample.

APPARATUS REQUIRED: Nessler’s cylinder, digital balance, beaker, pipette, measuring cylinder, glass rods.

CHEMICALS REQUIRED: Thioglycollic acid, standard ferric ammonium sulphate, distilled water, citric acid.

PRINCIPLE: The limit test is based on reaction between iron and thioglycollic acid in an alkaline media in the presence of 20% w/v of citric acid to produce ferrous thioglycollate which is deep red purple in colour.



Thioglycollic Acid Ferrous thioglycolate

20%w/v of citric acid is added which will prevent reaction between iron and NH_3 by forming a complex with it. Ammonia is added to provide alkalinity to the solution.

PROCEDURE:

Preparation of standard iron solution: Dissolve 0.173 gm of ferric ammonium sulphate in 10ml of 0.1 H_2SO_4. Add sufficient water to produce 1000 ml of solution. Each ml of solution contains 0.02mg of Fe.

Preparation of test solution: Weigh specific quantity of substance as specified in monograph and dissolve it in distilled water. Add 2 ml of 20 % w/v of citric acid and 0.1ml of thioglycollic acid and make the solution alkaline with NH_3 solution. And dilute it to 50ml with water and stir it well and allow it to stand for 5 min.

Preparation of standard solution: Take 2 ml of standard iron solution in Nessler’s cylinder and make up the volume to 40ml with distilled water and add 2 ml of 20% w/v solution of citric acid and 0.1ml of thioglycollic acid. Make the solution alkaline with ammonia and finally dilute to 50 ml with distilled water. Stir well and allow it to stand for 5 min. Compare the colour produced by the test solution by viewing test solution with standard solution by viewing transversely.

Summary:

Sample:	Observation	Inference
Sample(1gm) is dissolved in specific amount of water and then volume is made up to 40 ml+ 2 ml of 20 % w/v of citric acid (iron free) + 2 drops of thioglycollic acid+ ammonia 50 ml of water. Keep aside for 5 min.	The purple color produced in sample solution is greater than / less than standard solution.	The sample **passes/ doesn't pass** the limit test for iron.
Standard: Take 2 ml of standard solution of iron diluted with water upto 40ml + 2 ml of 20 % w/v of citric acid (iron free) + 2 drops of thioglycollic acid + ammonia + 50 ml water. Keep aside for 5 min		

OBSERVATION:

REPORT: The given sample______________the limit test for iron.

VIVA QUESTIONS:

1. Write the principle involved in limit test for Iron.
2. Write reaction involved in limit test for Iron.
3. The standard solution is prepared with________.
4. What is the importance of ammonia in limit test for Iron?
5. What is the agent used to produce colour in limit test for Iron?
6. On what criteria the standard and test solutions are compared?
7. What is the purpose of 20%w/v of citric acid?
8. What is the importance of thioglycollic acid in limit test for Iron?
9. What type of colour produced in limit test for Iron?

EXPERIMENT - 7

LIMIT TEST FOR HEAVY METALS

AIM: To perform limit test for heavy metals in given sample.

APPARATUS REQUIRED: Nessler's cylinder, pipette, beaker, measuring cylinder and glass rod.

CHEMICALS REQUIRED: Dil. CH_3COOH, dil. NH_3, HCl solution, H_2S solution, pH paper and standard lead solution.

PRINCIPLE: Limit test of heavy metals is based on the reaction of metallic impurities with hydrogen sulfide in acidic medium to form brownish colour solution. Metals that respond to this test are lead, mercury, bismuth, arsenic, antimony, tin, cadmium, silver, copper, and molybdenum. The metallic impurities in substances are expressed as parts of lead per million parts of the substance. The usual limit as per Indian Pharmacopoeia is 20 ppm

Dilute CH_3COOH and dil NH_3 solution is used to maintain pH between 3 to 4 so that the precipitate formed is colloid and uniform. The saturated solution of hydrogen sulphide should be prepared freshly for the experiment.

PROCEDURE:

Preparation of standard solution:

Take 2 ml of standard lead solution and dilute to 25 ml with water in Nessler's cylinder and add CH_3COOH (dil) or dilute NH_3 solution to adjust the p^H between 3 to 4 and then dilute the above solution with water upto 35 ml and add freshly prepared 10 ml of hydrogen sulphide solution & dilute with water to 50ml and allow to stand it for 5min.

Preparation of test solution:

Dissolve given sample in 25ml of water and transfer it to a Nessler's cylinder and add dil CH_3COOH or dil.NH_3 solution and just adjust the p^H between 3-4 and make up the volume to 35 ml with water and add 10ml freshly prepared hydrogen sulphide solution and finally make up the volume to 50ml with water and allow it to stand for 5min.

Summary:

Test:	Observation	Inference
20 ml of sample solution in Nessler’s cylinder + adjust the pH 3-4 with acetic acid or ammonia + 35 ml with water+ 10ml of hydrogen sulphide solution + Mix & set aside for 5 min.	The color produce in sample solution is **greater than/ less than** standard solution.	The sample ________ the limit test for Heavy metals.
Standard: Take 2ml of standard lead solution+ adjust the pH 3-4 with acetic acid or ammonia + 35 ml with water+ 10ml of hydrogen sulphide solution + Mix & set aside for 5 min.		

OBSERVATION:

REPORT: The given sample______________the limit test for heavy metals.

VIVA QUESTIONS:

1. Write the principle involved in limit test for Heavy metals.
2. Write reaction involved in limit test for Heavy metals.
3. The standard solution is prepared with________.
4. What are heavy metals?
5. What is importance of dilute acetic acid and dilute ammonia?
6. What pH range should be maintained in limit test for Heavy metals?
7. What gives colour to the solution?
8. Based on what criteria the standard and test are compared?

EXPERIMENT - 8

LIMIT TEST FOR LEAD

AIM: To perform limit test for heavy metals in given sample.

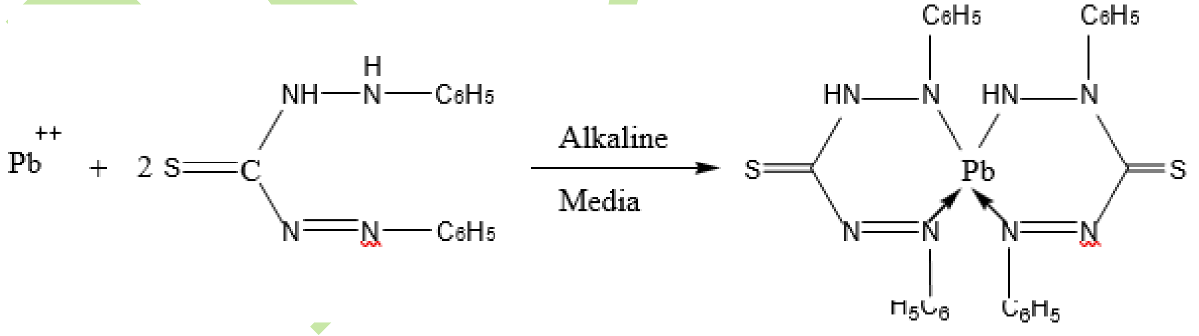
APPARATUS REQUIRED: Nessler's cylinder, pipette, beaker, measuring cylinder and glass rod.

CHEMICALS REQUIRED: Ammonium citrate, Potassium cyanide, standard lead solution, Hydroxylamine hydrochloride, Phenol red, Dithizone, Ammonia, Ammonium cyanide

Principle:

Limit test of lead is based on the reaction between lead and diphenyl thiocabazone (dithizone) in alkaline solution to form lead-dithizone complex which isred in color. The original dithizone has a green colour in chloroform thus the lead- dithizone shows violet colour. The intensity of the colour of complex is dependent upon the amount of lead in the solution. The colour of the lead-dithizone complex in chloroform has been compared with a standard lead solution, treated in the same manner. In this method, the lead present as an impurity in the substances, is separated by extracting an alkaline solution with a dithizone extraction solution. The interference and influence of other metal ion etc., is eliminated by adjusting the optimum pH for the extraction, by using ammonium citrate, potassium cyanide, hydroxylamine hydrochloride reagents, etc.

Reactions:



Procedure:

Test	Standard
A known quantity of sample solutionis transferred in a separating funnel	A standard lead solution is prepared equivalent to the amount of lead permitted in the sample under examination
Add 6 mL of ammonium citrate	Add 6 mL of ammonium citrate
Add 2 mL of potassium cyanide and 2 mL of hydroxylamine hydrochloride	Add 2 mL of potassium cyanide and 2 mL of hydroxylamine hydrochloride
Add 2 drops of phenol red	Add 2 drops of phenol red
Make solution alkaline by adding ammonia solution.	Make solution alkaline by adding ammonia solution.
Extract with 5 mL of dithizone until it becomes green	Extract with 5 mL of dithizone until it becomes green
Combine dithizone extracts are shaken for 30 sec. with 30 mL of 1.0% nitric acid and the chloroform layer is Discarded	Combine dithizone extracts are shakenfor 30 sec. with 30 mL of 1.0% nitric acid and the chloroform layer is discarded
To the acid solution add 5 mL of standard dithizone solution	To the acid solution add 5 mL of standard dithizone solution
Add 4 mL of ammonium cyanide	Add 4 mL of ammonium cyanide
Shake for 30 sec.	
Observe the colour developed	

Note:

1. All reagents and solutions used in the test must be free fromlead.
2. **Lead Standard Solution**: On the day of use, dilute 10 mL of lead nitrate stock solution with water to 100 mL. A control comparison solution prepared with 2.0 mL of lead standard solution contains, when compared to a solution representing 1.0 g of the substance under examination, the equivalent of 20 ppm lead.
3. **Lead Nitrate Stock Solution**: Dissolve 0.1598 g of lead nitrate in 100 mLof water to which has

been added 1 mL of nitric acid, then dilute to 1000 mL with water.

4. Lead has been one of the most undesirable impurities in medicinal substances. The chief sources of this impurity have been the sulfuric acid and the lead-lined apparatus. Besides, glass bottles used for storage of chemicals have been dangerous sources of contamination with lead.
5. The colour in lead limit test has been due to colloidal particles of lead sulphide and has been governed by the degree of fineness and degree of dispersion of these particles. The dispersion of particles could be controlled by the magnitude of electric charges which are carried by them. Addition of other substances modified the charges on the particles and their dispersion, thereby altering intensity of the original colour. That is why it is usual to use two solutions in the Lead Limit Test, both having original substance in solution.
6. Phenol red is used as indicator to develop the color at the end of process
7. The colour developed in the sample and standard solution is compared by keeping the Nessler"s cylinder against dark background and observing side by side.

OBSERVATION:

REPORT: The given sample______________the limit test for lead.

VIVA QUESTIONS:

1. Write the principle involved in limit test for Lead.
2. Write reaction involved in limit test for Lead.
3. The standard solution is prepared with________.
4. What gives colour to the solution?
5. Based on what criteria the standard and test are compared?

EXPERIMENT - 9

LIMIT TEST FOR ARSENIC

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

APPARATUS REQUIRED: Gutzeit apparatus, measuring cylinder, beaker and pipette.

CHEMICALS REQUIRED: Lead acetate cotton wool, mercuric chloride paper, zinc metal, stannous chloride, arsenic trioxide, potassium iodide, ammonium chloride etc.

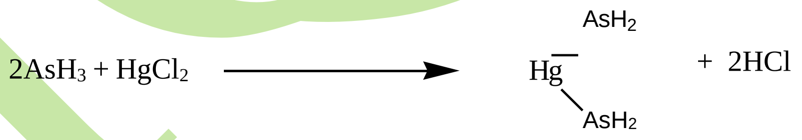
PRINCIPLE: The test is based on the fact that arsenic gets converted into arsenic acid in presence of an acid, which gets reduced by reducing agents (potassium iodide, stannous acid, zinc etc.,) to arsenious acid.

$$\underset{\text{Arsenic acid}}{H_3AsO_4} \xrightarrow{SnCl_2} \underset{\text{Arsenious acid}}{H_3AsO_3}$$

The nascent hydrogen produced by zinc and dil HCl reduced this arsenious acid to arsine gas.

$$H_3AsO_3 \xrightarrow[\text{Zn + dil HCl}]{3\ H_2} \underset{\text{Arsine gas}}{AsH_3} \uparrow + 3\ H_2O$$

This gas is passed over mercuric chloride paper, it produces a strain, which ranges from yellow to brown. The intensity and length are proportional to amount of arsenic.

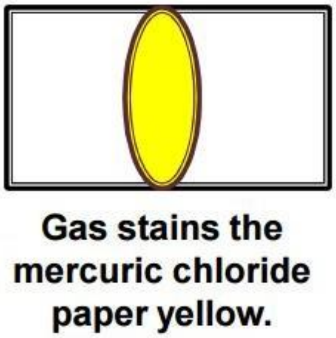


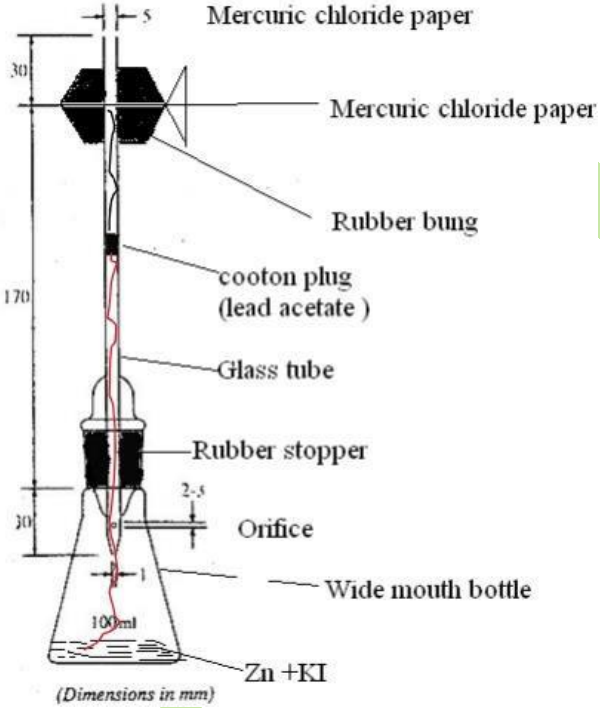
$$2AsH_3 + HgCl_2 \longrightarrow Hg(AsH_2)_2 + 2HCl$$

Mercuric Arsenide

The stain of the test sample is compared in day light with standard stains produced by known quantity of arsenic in the sample.

APPARATUS: A wide mouthed glass bottle capable of holding 120 ml is fitted to a rubber bung through which passes a glass tube, the tube made from ordinary glass has total length of 200 nm, internal diameter of 6.5 mm and external diameter of about 8 mm. It is drawn out at one end and to a diameter of 1 mm and a hole not less than 2 mm in diameter is blown in the side of the tube near constricted part. When rubber bung is inserted in the bottle containing 70 ml of liquid, the constricted end of the tube is above the surface of the liquid and hole in the side is below the bottom of the bung, upper end of the tube is a cut off square and is either slightly rounded or ground smooth.





PROCEDURE:

For standard solution: Add 1 gm of potassium iodide AsT and 10 gm of zinc dust AsT in a wide mouthed glass bottle. Then 10 ml of stannated HCl and 1 ml of dilute arsenic solution are added to the above solution. Then add 50 ml of water, glass tube is placed quickly in position. The glass tube is tightly placed with lead acetate cotton wool. The reaction is allowed to take place for 45 to 90 minutes. To accurate the reaction the apparatus is kept on hot surface for atleast 10 minutes.

For test solution: Add 1 gm of potassium iodide AsT and 10 gm of zinc dust AsT in a wide mouthed glass bottle. Then 10 ml of stannated HCl and 1 ml of test solution (2.5 gm of ammonium chloride in 50 ml of water) are added to the above solution. Glass tube is placed quickly in position. The glass tube is tightly placed with lead acetate cotton wool. The reaction is allowed to take place for 45 to 90 minutes. To accurate the reaction the apparatus is kept on hot surface for atleast 10 minutes.

Standard stain is compared with test stain. Limit not more than 4 ppm.

Preparation of reagents

Stannous chloride solution: Dissolve 330 g of stannous chloride in 100 ml of hydrochloric acid and add sufficient water to make 1000 ml.

Stannous chloride solution AsT: It is prepared by adding stannous chloride solution to an equal volume of hydrochloric acid AsT, reducing to the original volume by boiling and filtering through a fine-grain filter paper.

Stannated hydrochloric acid AsT: It is prepared by adding 1 ml of stannous chloride solution AsT to 100 ml of hydrochloric acid AsT.

Potassium iodide: Dissolve 16.6gm of KI in sufficient water to produce 100ml.

Standard arsenic solution (10 ppm As): Dissolve 0.330 g of arsenic trioxide in 5 ml of 2M sodium hydroxide and dilute to 250.0 ml with water. Dilute 1 volume of this solution to 100 volumes with water.

OBSERVATION:

REPORT: The given sample_________the limit test for arsenic.

VIVA QUESTIONS:

1. In which form the arsenic impurities are present?
2. What are the chemicals required for performing Limit test for Arsenic?
3. Write the principle involved in limit test for Arsenic.
4. Write the reactions involved in limit test for Arsenic.
5. What are different reducing agents used in limit test for Arsenic?
6. In what medium the limit test for Arsenic is performed?
7. How the nascent hydrogen is obtained in limit test for Arsenic?
8. What type of paper is used in limit test for Arsenic?
9. What is the apparatus used in limit test for Arsenic?
10. What colour of stain produced on mercuric chloride paper?
11. What forms stain on mercuric chloride paper?
12. How arsine gas is produced?

EXPERIMENT - 10

IDENTIFICATION TEST FOR MAGNESIUM HYDROXIDE

AIM: To carry out the identification tests with a given sample of Magnesium hydroxide

CHEMICALS REQUIRED: Ammonia solution, Ammonium chloride, Disodium hydrogen phosphate, NaOH, HCl, and Iodine.

APPARATUS REQUIRED: Beaker, pipette, stirrer, balance.

PROCEDURE:

S.No	Experiment	Observation	Inference
1	Dissolve about 15 mg of substance in 2 ml of water or use 2 ml of the prescribed solution. Add 1ml of dilute ammonia solution.	White precipitate is formed which is soluble 1 ml of 2 M ammonium chloride. White crystalline ppt reappears on adding 1 ml of 0.25 M disodium hydrogen phosphate	Presence of Magnesium
$Mg_2^+ + 2NH_3 + HPO_4^{2-} \longrightarrow Mg(NH_4)PO_4$ Magnesium ammonium phosphate			
2	Dissolve 10 mg in 1ml of HCl Add 2ml of sodium hydroxide	White gelatinous ppt is produces which is insoluble in excess of sodium hydroxide. Add few drops of iodine the ppt turns dark brown	Presence of Magnesium

REPORT:

Questions:

1. Give the chemical formula of Magnesium hydroxide.
2. Write the uses of Magnesium hydroxide.
3. Other name of magnesium hydroxide.
4. How do you identify magnesium ion?
5. How do you identify hydroxide ion?
6. What is the effect of magnesium compounds?

EXPERIMENT - 11

IDENTIFICATION TEST FOR FERROUS SULPHATE

AIM: To carry out the identification tests with a given sample of ferrous sulphate

CHEMICALS REQUIRED: Dil. Sulphuric acid, Phenanthroline, cerric ammonium sulphate, potassium ferricyanide, Dil.HCl.

APPARATUS REQUIRED: pipette, test tube, beaker, stirrer, and spatula.

PROCEDURE:

Ferrous ion:

S.No	Experiment	Observation	Inference
1.	Transfer 10 mg of substance into a test tube and add 2ml of water. Add 2 ml of dilute sulphuric acid and 1 ml of a 0.1 per cent w/v solution of 1,10-phenanthroline	Intense red colour which is discharged by addition of a slight excess of 0.1 M cerric ammonium sulphate	Presence of Ferrous
$Fe^{2+} + \text{Ligand} \longrightarrow \text{Complex}$ $Fe^{2+} + Ce^{4+} \longrightarrow Fe^{3+} + Ce^{3+}$			
2	Transfer 10 mg of substance into a test tube and add 2ml of water. add 1 ml of potassium ferricyanide solution	Dark blue precipitate is formed that is insoluble in dilute hydrochloric acid and is decomposed by sodium hydroxide solution.	Presence of Ferrous
$Fe^{2+} + 2K^{+} + [Fe(CN)_6]^{4}$ ----------- $K_2Fe[Fe(CN)_6]$			
3	Transfer 10 mg of substance into a test tube and add 1 ml of potassium ferrocyanide solution	a white precipitate is formed which rapidly becomes blue and is insoluble in dilute hydrochloric acid	Presence of Ferrous
$2\,FeSO_4 + K_4[Fe(CN)_6]$ ---------- $Fe_2[Fe(CN)_6]$ (white ppt) + SO_4^{2-} $Fe_2[Fe(CN)_6] \xrightarrow{(O)} Fe_4[Fe(CN)_6]_3$ (blue ppt)			

Sulphate ion:

S.No	Experiment	Observation	Inference
1	Transfer 50mg of test substance and add 5ml of water. Add 1ml of dilute hydrochloric acid and 1 rnl of barium chloride solution	White precipitate	Presence of sulphates
$SO_4^{2-} + BaCl_2$-------------------------$BaSO_4$ (ppt) + $2Cl^-$			
2	Add 0.1 rnl of iodine solution to the suspension obtained in the above test the suspension remains yellow (distinction from sulphites and dithionites) but is decolorised by adding, dropwise, stannous chloride Solution	No coloured precipitate	Presence of sulphates
3	Dissolve about 50 mg of the substance under examination in 5 rnl of water. Add 2 rnl of lead acetate solution	White precipitate, soluble in ammonium acetate solution and in sodium hydroxide solution	Presence of sulphates
SO_4^{2-} + $(CH_3COO)_2Pb$ ---------------$PbSO_4$ (ppt) + $2CH_3COONa$			

<u>REPORT:</u>

Questions:

1. Give the chemical formula of ferrous sulphate.
2. What is the synonym of ferrous sulphate
3. Write the uses of ferrous sulphate
4. How do you identify ferrous ion?
5. How do you identify sulphate ion?

EXPERIMENT - 12

IDENTIFICATION TEST FOR SODIUM BICARBONATE

AIM: To carry out the identification tests with a given sample of sodium bicarbonate

CHEMICALS REQUIRED: Phenolphthalein, potassium carbonate, potassium antimonite, magnesium uranyl acetate, acetic acid, magnesium sulphate, mercuric chloride.

APPARATUS REQUIRED: Bunsen burner, pipette, test tube, beaker, balance, water bath, stirrer, nichrome wire, conical flask.

PROCEDURE: **Sodium ion:**

S.No	Experiment	Observation	Inference
1	A little of the metal salt or other compound is mixed with a few drops of concentrated hydrochloric acid and a sample of the mixture is heated strongly in a bunsen flame on the end of a cleaned nichrome wire	Golden yellow colour	Presence of Sodium
2	Dissolve 0.1 g of the substance under examination in 2 rnl of water or use 2 rnl of the prescribed solution. Add 2 rnl of 15 per cent w/v solution of potassium carbonate and heat to boiling; no precipitate is produced. Add 4 rnl of a freshly prepared potassium antimonate solution and heat to boiling. Allow to cool in ice and if necessary, scratch the inside of the test-tube with a glass rod.	Dense, white precipitate	Presence of Sodium
$Na^{+} + KH_2SbO_4 \text{ ------------------ } NaH_2SbO_4 \text{ (ppt)} + K^{+}$			
3	Acidify a solution of the substance (0.1g) in 2ml water with 1M acetic acid and add a large excess of magnesium uranyl acetate solution	Yellow crystalline precipitate	Presence of Sodium
$Na^{+}+CH_3COOH + 3UO_2(CH_3COO)_2+ Mg(CH_3COO)_2+9\ H_2O \text{ ------ } NaMg(UO_2)_3(C_2H_3O_2)_9 .9H_2O + H^{+}$			

Bicarbonates ion:

<table>
<tr><th>S.No</th><th>Experiment</th><th>Observation</th><th>Inference</th></tr>
<tr><td>1</td><td>Treat a solution of the substance under examination with a solution of magnesium sulphate</td><td>No precipitate is formed (distinction from carbonates); boil, a white precipitate is formed.</td><td>Presence of Bicarbonates</td></tr>
<tr><td colspan="4">$HCO_3^- + Mg^{2+}$ ------------ $MgCO_3 + H_2O$</td></tr>
<tr><td>2</td><td>Introduce into a test-tube 0.1 g of the substance under examination suspended in 2 ml of water or in 2 ml of the prescribed solution Add 2 ml of 2 M acetic acid, close the tube immediately using a stopper fitted with a glass tube bent at two right-angles, heat gently and collect the gas in 5 ml of barium hydroxide solution</td><td>White precipitate forms that dissolve on addition of an excess of dilute hydrochloric acid.</td><td>Presence of Bicarbonates</td></tr>
<tr><td colspan="4">$HCO_3^- + CH_3COOH$ ------ $CH_3COO^- + H_2O + CO_2$
$CO_2 + Ba(OH)_2$ --------- $BaCO_3$ (ppt) $+ H_2O$
$BaCO_3 + 2\ HCl$ ------- $BaCl_2 + H_2O + CO_2$</td></tr>
<tr><td>3</td><td>Aqueous solution of bicarbonates is treated with mercuric chloride solution.</td><td>White Precipitate is formed which changes colour to brownish red colour.</td><td>Presence of Bicarbonates</td></tr>
<tr><td colspan="4">$2NaHCO_3 + HgCl_2$ ----------- $Hg(HCO_3)_2 + 2NaCl$
$Hg(HCO_3)_2$ ------ $Hg(OH)_2 + 2CO_2 \uparrow$
$Hg(OH)_2$ --------- $HgO + H_2O$</td></tr>
</table>

REPORT:

Questions:

1. Give the chemical formula of sodium bicarbonate.
2. What is the synonym of sodium bicarbonate?
3. Write the uses of sodium bicarbonate.
4. How do you identify sodium ion?
5. How do you identify bicarbonate ion?

EXPERIMENT - 13

IDENTIFICATION TEST FOR CALCIUM GLUCONATE

AIM: To carry out the identification tests with a given sample of calcium gluconate

CHEMICALS REQUIRED: Conc. HCl, potassium ferrocyanide, acetic acid, ammonium chloride, ammonium oxalate, sodium hydroxide, ammonium carbonate, phenyl hydrazine, ferric chloride.

APPARATUS REQUIRED: Bunsen burner, pipette, test tube, beaker, balance, water bath, stirrer, nichrome wire, conical flask, melting point apparatus.

PROCEDURE: Calcium ion:

S.No	Experiment	Observation	Inference
1	A little of the metal salt or other compound is mixed with a few drops of concentrated hydrochloric acid and a sample of the mixture is heated strongly in a bunsen flame on the end of a cleaned nichrome wire	Brick red flame is produced	Presence of calcium
2	Dissolve 20 mg of the substance under examination in 5 ml of 5M acetic acid. Add 0.5 ml of potassium ferrocyanide solution, the solution remains clear. Add about 50 mg of ammonium Chloride	White crystalline precipitate	Presence of calcium
$2Ca^{2+} + K_4[Fe(CN)_6 + 4NH_4Cl$ ------------------------ $2(NH_4)_2 Ca (Fe(CN)_6)$ (ppt) + $4KCl + CaCl_2$			
3	To 5 ml of a 0.4 per cent w/v solution of the substance under examination add 0.2 ml of a 2 per cent w/v solution of ammonium oxalate.	white precipitate is obtained that is only sparingly soluble in dilute acetic acid but is soluble in hydrochloric acid.	Presence of calcium
$Ca^{2+} + (COO)_2 (NH_4)_2$--------------------$Ca(COO)_2$ (ppt)			
4	Dissolve 20 mg of the substance under examination in the minimum quantity of dilute hydrochloric acid and neutralise with dilute sodium hydroxide solution. Add 5 ml of ammonium carbonate solution	White precipitate is formed which, after boiling and cooling the mixture, is only sparingly soluble in ammonium chloride solution	Presence of calcium
$Ca^{2+} + (NH_4)_2CO_3$--------------$CaCO_3 + NH_4Cl$			

5	To 1 ml of sample add 1 drop of ferric chloride	Dark yellow colour	Presence of calcium
6	Flame test:	Yellow red colour	Presence of calcium

Gluconic Acid:

S.No	**Experiment**	**Observation**	**Inference**
1	To 1 ml of a 3 per cent w/v solution add 0.05ml of ferric chloride test solution	yellow colour	Presence of Gluconic acid
2	Dissolve 0.75g of sample in 7.5ml of water and add 1ml of glacial acetic acid, 1.5ml phenyl hydrazine. Heat for half an hour on a water bath and cool. Scratch the inner surface of the test tube with a glass rod until crystals of gluconic acid phenyl hydrazine begins to form. Keep the test tube for 10 min. Filter. Dissolve the precipitate in 10ml of hot water. Add small amount of charcoal and filter. Allow to cool to recover the white crystals	Product melts at 200°C with decomposition	Presence of Gluconic acid
$HOCH_2(CHOH)_4COOH + C_6H_5NHNH_2$ ------------ $HOCH_2(CHOH)_4CO\ NHNH\ C_6H_5$(Gluconic acid phenyl hydrazide)			

REPORT:

Questions:

1. Give the chemical formula of calcium gluconate.
2. Write the uses of calcium gluconate.
3. How do you identify calcium ion?
4. How do you identify gluconate ion?
5. What is the effect of calcium compounds?

EXPERIMENT - 14

IDENTIFICATION TEST FOR COPPER SULPHATE

AIM: To carry out the identification tests with a given sample of copper sulphate.

CHEMICALS REQUIRED: Conc. hydrochloric acid, ammonia, sodium hydroxide, barium chloride, iodine, stannous chloride, lead acetate, ammonium acetate, sodium hydroxide.

APPARATUS REQUIRED: Bunsen burner, nichrome wire, test tube, beaker, glass rod, pipette,

PROCEDURE:

Copper ion:

S.No	Experiment	Observation	Inference
1	**Flame test** A little of the metal salt or other compound is mixed with a few drops of conc. hydrochloric acid and a sample of the mixture is heated strongly in a bunsen flame on the end of a cleaned nichrome wire	Vivid blue flame	Presence of Copper
2	Few mg of sample is treated with ammonia or sodium hydroxide	Blue/turquoise ppt. of copper(II) hydroxide which dissolves in excess ammonia to give a deep blue solution of an ammine complex, but copper(II) hydroxide is NOT soluble in excess NaOH.	Presence of Copper
$Cu^{2+}{}_{(aq)} + 2OH^{-}{}_{(aq)} ==> Cu(OH)_{2(s)}$ $Cu(OH)_{2(s)} + 4NH_{3(aq)} ==> [Cu(NH_3)_4]^{2+}{}_{(aq)} + 2OH^{-}{}_{(aq)}$			

Sulphate ion:

S.No	Experiment	Observation	Inference
1	Transfer 50mg of test substance and add 5ml of water. Add 1ml of dilute hydrochloric acid and 1 ml of barium chloride solution	white precipitate	Presence of Sulphates
$SO_4^{2-} + BaCl_2$ -------------------------- $BaSO_4$ (ppt) + $2Cl^-$			
2	Add 0.1 rnl of iodine solution to the suspension obtained in the above test the suspension remains yellow (distinction from sulphites and dithionites) but is decolorised by adding, dropwise, stannous chloride Solution	No coloured precipitate	Presence of Sulphates
3	Dissolve about 50 mg of the substance under examination in 5 rnl of water. Add 2 rnl of lead acetate solution	white precipitate, soluble in ammonium acetate solution and in sodium hydroxide solution	Sulphates
SO_4^{2-} + $(CH_3COO)_2Pb$ --------------- $PbSO_4$ (ppt) + $2CH_3COONa$			

REPORT:

Questions:

1. Give the chemical formula of copper sulphate.
2. What is the synonym of copper sulphate?
3. Write the uses of copper sulphate.
4. How do you identify copper ion?
5. How do you identify sulphate ion?

EXPERIMENT - 15

SWELLING POWER IN BENTONITE

AIM: To determine swelling power in Bentonite.

APPARATUS REQUIRED: Graduated measuring cylinder.

CHEMICALS REQUIRED: Bentonite and Sodium lauryl sulphate.

PRINCIPLE: Bentonite is a colloidal hydrated aluminum silicate. It occurs as a very fine powder. It is odorless, pale buff or cream colored. Bentonite is slightly earthy in taste and hygroscopic. It is insoluble in water but swells approximately about 12 times its volume when added to water. Bentonite is insoluble and does not swell in organic solvents. The suspension of Bentonite in water has a pH between 9 to 10 particle size of Bentonite is 44 microns.

PROCEDURE: Weigh 2g of Bentonite in 20 portions and add at 2-minute intervals to 100 ml of 1% of sodium lauryl sulphate in a 100 ml graduated cylinder. Allow each portion to settle before adding the next and allow it to stand for 2 hrs. Then the apparent volume of sediment at bottom of cylinder shouldn't be less than 24 ml.

REPORT:

The swelling power of Bentonite was found to be_________ml.

VIVA QUESTIONS:

1. Define swelling power.
2. What are the uses of Bentonite?
3. Define suspension.
4. What is the use of SLS?
5. What are the physical properties of Bentonite?

EXPERIMENT - 16

ACID NEUTRALISING CAPACITY OF ALUMINIUM HYDROXIDE GEL

AIM: To determine acid neutralization capacity of aluminum hydroxide gel.

APPARATUS REQUIRED: Conical flask burette, burette stand and measuring cylinder.

CHEMICALS REQUIRED: Aluminum hydroxide gel, 0.1N HCl, and Bromophenol blue and Sodium hydroxide.

PRINCIPLE: Aluminum hydroxide gel is an aqueous white viscous suspension of hydrated aluminum oxide having varying amounts of basic aluminum carbonate. In general, it is tested for the presence of acid consuming capacity by titrating the excess of acid in aluminum hydroxide gel by sodium hydroxide solution.

PROCEDURE: Place 1.5 ml of aluminum hydroxide gel in conical flask to it add 50 ml of 0.1.N HCl and shake the contents of the flask at 37°C for one hour then the solution is titrated with 0.1N sodium hydroxide solution using bromophenol blue as an indicator.

NOTE: 1gm of gel should consume not less than 12.5ml and not more than 25ml of 0.1N HCl.

REPORT:

The amount of acid consumed by aluminum hydroxide gel was found to be_________ml.

VIVA QUESTIONS:

1. What is aluminum hydroxide gel?
2. Define antacid.
3. What is acid consuming capacity?
4. How u will determine the acid consuming capacity?
5. How aluminum hydroxide gel was prepared?
6. What is the need to identify the acid consuming capacity?

EXPERIMENT - 17

PRESENCE OF IODATE IN POTASSIUM IODIDE

AIM: To Perform test for purity for potassium iodide by examining the presence of iodates in the sample.

APPARATUS: Test tubes, measuring jar, colour comparision tube.

CHEMICALS REQUIRED: Potassium iodide, dil H_2SO_4, iodide free starch solution, concentrated ammonia, standard iodate solution.

PRINCIPLE:

Iodates are the impurities present in potassium iodide by preparing the standard solution using standard iodate solution and starch and also by preparing test solution the colour produced in test and standard is compared with the iodide ions starch will combines to give blue colour

PROCEDURE

TEST-1: Dissolve 0.5gm of potassium iodide in 10ml of carbon dioxide free water and add 0.15ml of dil H_2SO_4 and one drop of iodide free starch solution if no blue color is produced with in 2min it indicates the presence of iodate ions.

TEST-2: Dissolve 1.1gm of potassium iodide in sufficient ammonia and carbon dioxide free water to yield 10ml of solution and transfer to a colour comparison tube. Add 1ml of starch solution, 0.25ml of $1NH_2SO_4$ mix and compare the colour with that of the colour of similar volume of standard solution.

PREPARATION OF STANDARD SOLUTION:

100mg of potassium iodide, 1ml of standard iodate solution,1ml of starch and 0.25ml of 1N H_2SO_4 are added in a test tube. The colour produced in this test is compared with the test solution the test specimen colour does not exceeded than the colour produced in the standard solution (4ppm)

Report: Test for purity for given sample of KI was performed and iodate ions ---------------------

EXPERIMENT - 18

PREPARATION OF BORIC ACID

AIM: To prepare and submit boric acid.

APPARATUS REQUIRED: Conical flask, beaker, measuring cylinder, funnel etc

CHEMICALS REQUIRED: 20% Sulfuric acid, Borax, purified water, phenolphthalein

PRINCIPLE: Boric acid is also called as ortho boric acid or acidum boricum. It is a mild acid and is used as an anti-septic and as a precursor of other pharmaceutical compounds. It exists in the form of colorless crystals or a white powder. It has the chemical formula H_3BO_3. The preparation of boric acid involves simple principle where the borax reacts with sulfuric acid to form boric acid.

$$Na_2B_4O_7.10H_2O.10H_2O+H_2SO_4 \rightarrow H_3BO_3+Na_2SO_4+6H_2O$$

PROCEDURE: Accurately weigh about 5 gm of borax and dissolve in 50 mL of purified water, and add 1-2 drops of phenolphthalein which gives pink color to the solution. Then add 20% sulfuric acid slowly with constant stirring until the total reaction mixture is colorless. Then transfer the mixture into a china dish and heat the solution in a low flame until the solution is completely evaporated. Then set the mixture aside for crystallization. Then the crystals are dried at room temperature.

USES: Boric acid is used as anti-infective, antiseptic for burns and cuts and is also used in eye drops (1.5% as an eye wash)

REPORT: Boric acid was prepared and submitted. The percentage yield of boric acid was found to be ______.

VIVA QUESTIONS:

1. Define antiseptic.
2. Define anti-infective.
3. Write the reaction involved the preparation of boric acid?
4. What are uses of boric acid?
5. What is chemical formula of boric acid?
6. What are synonyms of boric acid?

EXPERIMENT - 19

PREPARATION OF POTASH ALUM

AIM: To prepare and submit potash alum.

APPARATUS REQUIRED: Beaker, measuring cylinder, glass rod, china dish, funnel

CHEMICALS REQUIRED: Aluminium sulphate, Potassium sulphate, distilled water

PRINCIPLE: Alum is a double salt, univalent and trivalent metal salt with 24 moles of water per mole. Alum is prepared by reaction between aluminium sulphate and potassium sulphate.

$$K_2SO_4 + Al_2(SO_4)_3.16\,H_2O + 8H_2O \rightarrow K_2SO_4.Al_2(SO_4)_3.24\,H_2O$$

PROCEDURE: Weigh accurately 1.25 gm of Potassium sulphate and 4.25 gm of Aluminium sulphate and dissolve them in a minimum quantity of water in different beakers. Heat both the solutions simultaneously and then add hot potassium sulphate solution to hot aluminium sulphate solution. Then the solution obtained is evaporated until the volume is reduced to 3/4th of its original volume. Then it is set aside for crystallization.

USES: Potash alum is used as astringent and antiseptic.

REPORT: Potash alum was prepared and submitted. The percentage yield of potash alum was found to be______.

VIVA QUESTIONS:

1. What is the chemical name of potash alum?
2. What is the composition of potash alum?
3. How is potash alum prepared?
4. What are the uses of alum?
5. Define antiseptic.
6. Define astringent.

EXPERIMENT - 20

PREPARATION OF FERROUS SULPHATE

AIM: To prepare and submit ferrous sulphate

APPARATUS REQUIRED: Beaker, measuring cylinder, glass rod, china dish, funnel

CHEMICALS REQUIRED: Iron filings, dil. Sulphuric acid, distilled water

PRINCIPLE: Ferrous sulphate is also called as green vitriol. It is bluish green in colour, crystalline, efflorescent in air. On exposure to air the crystals rapidly oxidize and become brown. Ferrous sulphate is prepared by treating iron filings or powder with dilute or concentrated sulphuric acid.

$$Fe + H_2SO_4 \longrightarrow FeSO_4 + H_2 \uparrow$$

PROCEDURE: Take 25ml of dilute sulphuric acid in a beaker and boil. Add iron filings in small portions with gradual heating until effervescence of H_2 ceases. Filter the solution and allow it to cool. The crystals obtained are separated, dried and recrystallized again using distilled water.

USES: Ferrous sulphate is used as hematinic and in the treatment of Iron deficiency anemia.

REPORT: Ferrous sulphate was prepared and submitted. The percentage yield of ferrous sulphate was found to be_______.

VIVA QUESTIONS:

1. What is the chemical name of ferrous sulphate?
2. Write the chemical reaction involved in the preparation of ferrous sulphate.
3. What are the uses of ferrous sulphate?
4. Define hematinic?

EXPERIMENT - 21

PREPARATION OF MAGNESIUM SULPHATE

AIM: To prepare and submit magnesium sulphate

APPARATUS REQUIRED: Beaker, measuring cylinder, glass rod, china dish, funnel

CHEMICALS REQUIRED: Magnesium oxide, dil. Sulphuric acid, distilled water

PRINCIPLE: It is often a heptahydrate sulphate and commonly called Epsom salt. It is prepared from magnesium oxide. the oxide is reacted with sulfuric acid to produce magnesium sulphate

$$MgO(s) + H_2SO_4(aq) \rightarrow MgSO_4(aq)$$

Magnesium sulfate, $MgSO_4.7H_2O$, crystals are obtained by evaporation. They may be recrystallised to increase purity.

PROCEDURE: Weight out about 1.5 g magnesium oxide. Using a measuring cylinder, measure 25 ml of 1 mol sulfuric acid taken in a beaker. Warm the acid to about 60 °C and, while stirring the acid, add magnesium oxide a little at a time. Make sure each portion dissolves before adding more. After about 1g no more will dissolve and you will see a cloudy suspension in the beaker. evaporate slowly over a hot water bath at about 60 °C until crystals form.

USES: Laxative

REPORT: Magnesium sulphate was prepared and submitted. The percentage yield of magnesium sulphate was found to be_______.

VIVA QUESTIONS:

1. What is the chemical name of magnesium sulphate?
2. What is the synonym of magnesium sulphate?
3. Write the chemical reaction involved in the preparation of magnesium sulphate.
4. What are the uses of magnesium sulphate?
5. Define laxative?