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

B. PHARM 1st SEMESTER

EAST POINT COLLEGE OF PHARMACY
East Point Campus, Jnana Prabha, Virgo Nagar Post,
Bengaluru – 560049, Karnataka

B Pharmacy

Program Outcomes (PO's)

PO 1- Pharmacy Knowledge

Possess knowledge and comprehension of the core and basic knowledge associated with the profession of pharmacy, including biomedical sciences; pharmaceutical sciences; behavioral, social, and administrative pharmacy sciences; and manufacturing practices.

PO 2- Planning Abilities

Demonstrate effective planning abilities including time management, resource management, delegation skills and organizational skills. Develop and implement plans and organize work to meet deadlines.

PO 3- Problem analysis

Utilize the principles of scientific enquiry, thinking analytically, clearly and critically, while solving problems and making decisions during daily practice. Find, analyze, evaluate and apply information systematically and shall make defensible decisions

PO 4- Modern tool usage

Learn, select, and apply appropriate methods and procedures, resources, and modern pharmacy-related computing tools with an understanding of the limitations.

PO 5- Leadership skills

Understand and consider the human reaction to change, motivation issues, leadership and team-building when planning changes required for fulfillment of practice, professional and societal responsibilities. Assume participatory roles as responsible citizens or leadership roles when appropriate to facilitate improvement in health and wellbeing.

PO 6- Professional Identity

Understand, analyse and communicate the value of their professional roles in society (e.g. health care professionals, promoters of health, educators, managers, employers, employees).

PO 7- Pharmaceutical Ethics

Honor personal values and apply ethical principles in professional and social contexts. Demonstrate behaviour that recognizes cultural and personal variability in values, communication and lifestyles. Use ethical frameworks; apply ethical principles while making decisions and take responsibility for the outcomes associated with the decisions

PO 8- Communication

Communicate effectively with the pharmacy community and with society at large, such as, being able to comprehend and write effective reports, make effective presentations and documentation, and give and receive clear instructions

PO 9- The Pharmacist and society

Apply reasoning informed by the contextual knowledge to assess societal, health, safety and legal issues and the consequent responsibilities relevant to the professional pharmacy practice.

PO 10- Environment and sustainability

Understand the impact of the professional pharmacy solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.

PO 11- Life-long learning

Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change. Self-access and use feedback effectively from others to identify learning needs and to satisfy these needs on an ongoing basis.

Programme Specific Outcomes (PSO's)	
PSO 1	Acquire a thorough foundational knowledge in pharmaceutical sciences, including pharmacology, pharmaceutics, medicinal chemistry, and pharmacognosy, to excel in further academic pursuits
PSO 2	Gain expertise in the application of contemporary pharmaceutical techniques and technologies, enhancing employability across various sectors including the pharmaceutical industry, academia, and research institutions.
PSO 3.	Equip with entrepreneurial skills and knowledge of pharmaceutical business management, including market analysis, product development, regulatory affairs, and financial planning, to initiate and run successful ventures in the pharmacy sector

Course Outcomes (CO's)
Code: BP110P Pharmaceutical Inorganic Chemistry
CO1- The level of specific impurities in the given inorganic compounds by performing different limit tests
CO2- Different chemical methods to prepare inorganic pharmaceuticals.
CO3- Perform identification tests as per Indian Pharmacopoeia
CO4- Determine the impurities qualitatively by performing tests for purity
CO5- Determine the Neutralizing capacity for antacids

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INTRODUCTION TO LIMIT TEST

Practically it is impossible to remove all the impurity from any substance. Some remain in 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 be tolerated in pharmaceutical drugs and excipients. The limit tests help to check and indicate the presence 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 with the standard taking the specified amount of impurity.

The standard substances of limit test contain the maximum amount of impurity which can 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

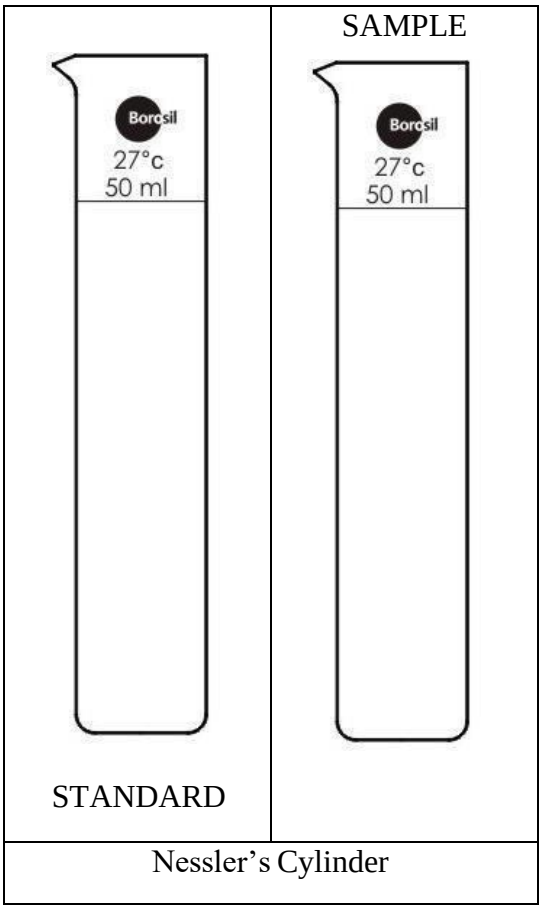
Cumulative impurity: The impurity which may not cause immediate effect, but when taken for long period of time shows toxicity. Example: Heavy metals

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 foreach 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. For harmless impurity the limits are still high.

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.

Reference: Indian Pharmacopoeia **2007, pg.: 76.**

Apparatus & Chemical Requirements - Nessler's cylinder, Glass rod, pipette, 5% silver nitrate solution, 0.05845%w/v sodium chloride, dilute nitric acid, distilled water.

Principle:

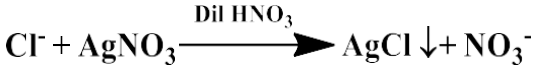
The limit test for chloride is based upon the reaction between soluble chloride and silver nitrate reagent in presence of dilute nitric acid to produce insoluble silver chloride.

The turbidity produce by the sample is compared with that of standard turbidity produce by the specific amount of chloride ions.

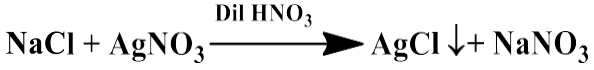
The sample turbidity should not be greater than the standard turbidity for passing the limit test for chloride.

Reaction:

Sample :



Standard :



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.

Reference: Indian Pharmacopoeia **2007, pg: 78**

Apparatus & Chemicals Required - Nessler's cylinder, Glass rod, pipette, 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 : $SO_4^- + BaCl_2 \xrightarrow{\text{Dil HCl}} BaSO_{4\downarrow} + 2Cl^-$

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

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.

Reference: Indian Pharmacopoeia, 2007, pg:77

Apparatus & Chemical Requirements- Nessler's cylinder, Glass rod, pipette, 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 mLof 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 strongreducing agent which reduce the Ferric ion to ferrous ion.

The color produce by the sample is compared with that of the Standard color producedby 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 thanthat 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 :

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

Ferrous Iron

Reasons

- Citric acid solution is added because it forms a soluble complex with iron and avoidsprecipitation as its hydroxide when Dil. ammonia solution is added.
- Dil. Ammonia solution is added to make the solution alkaline because ferrous thioglycolategives 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.

Reference: Indian Pharmacopoeia, 2007, pg: 76, 77

Apparatus & Chemical Requirements- Nessler's cylinder, Glass rod, pipette, hydrogen sulphide 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.

Reaction:

$$HM + H_2S \xrightarrow[H_2]{\text{Dil. } NH_3 \text{ soln}} HMS\downarrow +$$

$$PB^{2+} + H_2S \xrightarrow[+H^+]{\text{Dil } CH_3COOH/NH_3 \text{ Soln}} PbS\downarrow$$

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. 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. Add 10 mL freshly prepared hydrogen sulphide 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. 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. 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 morestable.
- The hydrogen sulphide solution is freshly prepared because on keeping the hydrogen sulphidegas 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 ^{o}C.
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 preparedH_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.

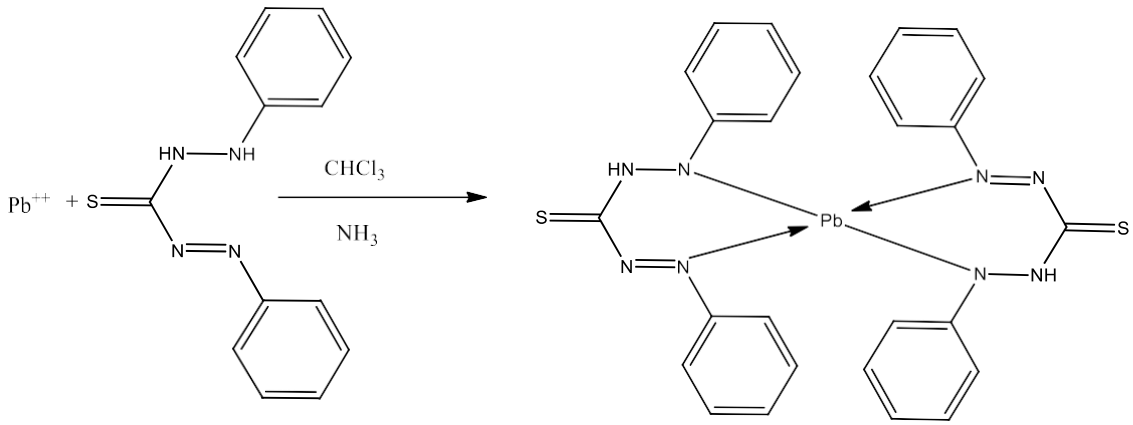
Reference: Indian Pharmacopoeia**,** 2007, pg: 77.

Apparatus & Chemical Requirements- Beaker, Glass rod, pipette, 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 ofother impurities.
- Dil. Ammonia solution is added to make the solution alkaline which will be indicated by phenolred 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

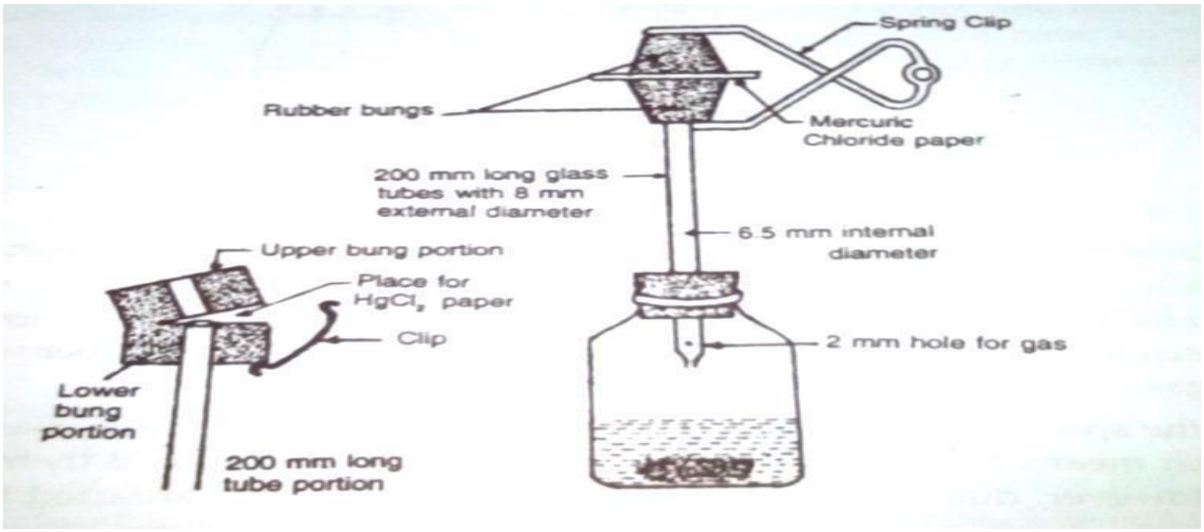
LIMIT TEST FOR ARSENIC

Aim - To carry out the limit test for arsenic for the given sample.

Reference: Indian Pharmacopoeia, 2007, pg: 76.

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 such a way that a mercuric chloride paper is sandwiched in between them. The two-rubber bung are held together tightly with a clip or a screw clamp. Inside the tube, 2 mm below the rubber bung lead acetate cotton is placed.



Gutzeit Apparatus

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 specificamount 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

$$H_3AsO_4 \xrightarrow{HCl / KI / SnCl_2} H_3AsO_3$$

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 rinsedwith 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 mercuricchloride 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 ^oC in dark.

Precautions

1. The apparatus must be properly washed with dilute HCl and the rinsed with water every timebefore and after the Arsenic limit test.
2. Mercuric chloride test paper must be freshly prepared by wetting the test paper in Mercuricchloride solution and drying under shade.
3. The delivery tube must not dip in to the mixture in the bottle, so that the Arsine gas allowedpassing through it.
4. The reaction must be allowed to proceed at 40 ^0C for 40min.
5. The standard strain and the sample strain should be prepare simultaneously and compareimmediately 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 convertsarsenic acid to arsenous acid.

2. Granulated zinc is added to produce the nascent hydrogen gas by reacting with HCl. Thenascent 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.

$$H_2S + HgCl_2 \longrightarrow HgS + 2HCl$$

$$H_2S + Pb(CH_3COO)_2 \longrightarrow \underset{\text{Black}}{PbS} + CH_3COOH$$

4. A side whole is provided in the delivery tube to allow continuous passage for arsine gas in thecase 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 sodium bicarbonate.

Reference: Indian pharmacopoeia; vol II, 2007, pg 1081

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

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

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?

Experiment No: 08

SPECIAL PROCEDURE FOR LIMIT TEST OF CHLORIDE AND SULPHATE FOR POTASSIUM PERMANGANATE

Aim: To perform special procedure for chloride and sulphate for potassium permanganate.

Reference: Indian pharmacopoeia 1996; vol III, 2007, pg 962.

Principle:

Potassium permanganate produces deep violet or purple colour solution in water which will mask the turbidity or turbidity produce by the sample. So boil the potassium permanganate with alcohol which convert the sample to MnO_2 precipitated and Potassium sulphate. The MnO_2 is filtered out and the filtrate is taken for limit test for chloride and sulphate.

Procedure:

Dissolve 1.5g of sample in 50 mL of distilled water, heat on water bath and add gradually 6 mL of ethanol (95 %), cool, dilute to 60 mL with distilled water and filtered. The filtrate is colorless and taken for limit test.

For Chloride

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	40 mL of above solution is taken in a clean Nessler's cylinder. Add 10 mL 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

For sulphate

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	10 mL of above solution is taken in a clean Nessler's cylinder. Add 2 mL of dilute HCland 5 mL of Barium Sulphate reagent. Make up the volume up to 50 mL by adding distilled water. Allow to stand for5min

Comparison

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

Observation & 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. Explain the principle of the limit test of chloride and sulphate in Potassium permanganate.
2. Why Potassium permanganate is boiled with alcohol before doing the limit test

Experiment No: 09

PREPARATION OF BORIC ACID

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

Reference: Text book of Inorganic pharmaceutical and medicinal chemistry by JS Qadry, pg: 74

Apparatus & Chemicals Required: Beaker 100 mL, glass rod, funnel, filter paper, borax, dil. Sulphuric acid, distilled water

Discussion:
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 = $\frac{6 \text{ X } (4 \text{ X } 61.83)}{381.24}$

= 3.891 g

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

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

Experiment No: 10

PREPARATION OF FERROUS SULPHATE

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

Reference: Chatwal G.R, Pharmaceutical Inorganic Chemistry

Apparatus & Chemicals Required: Beaker 100 mL, glass rod, funnel, filter paper, distilled water, Iron Fillings, Sulphuric acid.

Discussion:
Ferrous sulphate contains 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 isfiltered and allowed to cool.
- The crystals are separated and dried, then recrystallise from water.

Reaction:

$$FeSO_4 + H_2SO_4 \rightarrow FeSO_4 + H_2\uparrow$$

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: 11
PREPARATION OF POTASH ALUM

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

Reference: Text book of pharmaceutical chemistry - Inorganic by GR Chatwal, Page no.

Apparatus & Chemicals Required: Beaker 100 mL, glass rod, funnel, filter paper, distilled water, Potassium sulphate, aluminum sulphate.

Discussion:
Potash alum is prepared by dissolving an equimolar mixture of hydrated aluminium sulphate and potassium sulphate in minimum amount of water containing a little sulphuric acid and then subjecting the resulting solution to the crystallization, when octahydral crystals of potashalum 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 toit. 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

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

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

Experiment No:12

SWELLING POWER OF BENTONITE

Aim: To Study theswelling power of Bentonite

Reference: Textbook of pharmaceutical chemistry - Inorganic by GR Chatwal, Page no.

Apparatus & Chemicals Required: Graduated measuring cylinder, Bentonite, Sodium lauryl sulphate.

Discussion:

Bentonite is a natural colloidal, hydrated aluminum silicate that has been processed to removegrit and non-swelling components 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 a bulk laxative.
It is a pharmaceutical aid used as a suspending agent.
It can be used as 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 the sample passes the test for swelling power of bentonite.

Experiment No: 13

IDENTIFICATION OF CATIONS

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

Procedure:

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^+

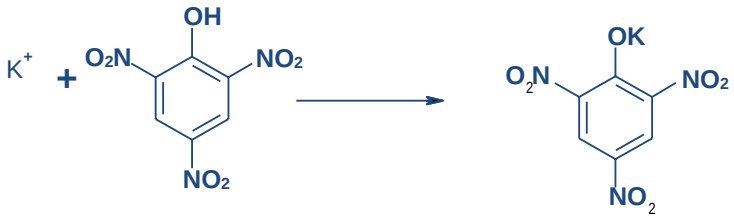
Test	Observation	Inference
To the sample solution add freshly prepare Sodium cobalt nitrate solution	Yellow ppt	K^+ ion present

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

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^{++} + (NH4)2CO3 □ CaCO3 + $NH4^{+}$

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

Ca^{++} + $(NH_4)_2(COO)_2$ → Ca $(COO)_2$ + 2 NH_4^{+}

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

Ca^{++} + K_2CrO_4 □ K^{+} + $CaCrO_4$

Identification for Fe^{++}

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

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

$4K_2Fe[Fe(CN)_6]_2 + 4H^{+} + O_2$ ⟶ $4KFe[Fe(CN)_6]$ + 4K + $2H_2O$

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

Experiment No: 14

IDENTIFICATION OF ANIONS

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

Procedure:

Identification for HCO_3^-

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

$$HCO3^- + HCl \longrightarrow H2O + CO2 + Cl^-$$

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

$$HCO3^- + MgSO4 \square Mg(HCO3)2 \square MgCO3 + H2O + CO2$$

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

$$HgCl2 + HCO3^- \square Hg(HCO3)2 + 2Cl^-$$

Identification for Cl^-

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

$$Cl^- + AgNO3 \xrightarrow{\text{Dil HNO3}} AgCl + NO3^-$$

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

$$K2Cr2O7 + 2\,Cl^- + 6\,H2SO4 \square 6\,HSO4^- + 3\,H2O +$$

$$Cr2O2Cl2\,Cr2O2Cl2 + H2O \square H2CrO4 + HCl$$

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

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

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

$$I^- + AgNO3 \xrightarrow{\text{dil HNO3}} AgI + NO3^-$$

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

$$2I^- + HgCl_2 \rightarrow 2Cl^- + HgCl_2$$
Red
$$HgCl_2 + 2KI \rightarrow HgI_4^-$$

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**.