



PRACTICAL LAB MANUAL

LAB MANUAL PHARMACEUTICAL INORGANIC
CHEMISTRY

B.PHARM Ist YEAR

LIST OF EXPERIMENTS

S. No.	EXPERIMENTS
1	To prepare and submit calcium carbonate (CaCO_3).
2	To prepare and submit magnesium carbonate (MgCO_3)
3	To prepare and submit Zinc sulphate (ZnSO_4).
4	To prepare and submit Potash alum ($\text{K}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 \cdot 24 \text{H}_2\text{O}$)
5	To prepare and submit Boric acid (H_3BO_3)
6	To prepare and submit aluminum hydroxide.
7	To perform limit test for chloride in given sample.
8	To perform limit test for sulphate in given sample.
9	To perform limit test for iron in given sample.
10	To perform identification test for boric acid.
11	To perform the identification test of ammonium chloride.
12	To identify cation & anion in given pharmaceutical compounds.

EXPERIMENT NO -1

OBJECT: To prepare calcium carbonate (CaCO₃).

REFERENCES

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed. Ist, 2008, pp 109.
2. Chatwal G.R, "Pharmaceutical chemistry inorganic" Himalaya publishing house, 5th Ed. 5th, 2010, pp 161-163.

REQUIREMENTS

Chemical requireds: Sodium carbonate (Na₂CO₃), Calcium carbonate (CaCl₂)

Apparatuue required: Beaker, funnel, glass rod & Measuring cylinder.

THEORY

Synonyms: Precipitated Chalk

Chemical formula: CaCO₃

Molecular weight 100.09

It has been regarded as one of the most abundant and widely distributed calcium salts. Calcium carbonate is found in nature as limestone, chalk, marble, argnite, and calcite and one of the main constituents of pearls and shells. It is a fine white odorless, tasteless microcrystalline powder. It is practically insoluble in water but its solubility is increased by the presence of any ammonium salts or carbon dioxide.

The official drug is the precipitated calcium carbonate containing 98 to 100.5 % of calcium carbonate. Hardness of water is due to its presence.

On commercial scale calcium carbonate is obtained by mixing the boiling solution of calcium chloride and sodium carbonate.



PROCEDURE

Take 3g of sodium carbonate (Na_2CO_3) and dissolved in 30 mL of distilled water in a 100mL beaker.

Now take 3g of calcium chloride (CaCl_2) and dissolved it in 30mL of water.

Then both the salts are mixed along with constant stirring.

Allowed to stand for 15 minutes, the precipitate of calcium carbonate is produced.

Solution is filtered with the help of filter paper. A white fine powder of calcium carbonate is formed as a residue on the filter paper and dry.

PROPERTIES

It is a fine white odorless, tasteless micro-crystalline powder.

It is practically insoluble in water but its solubility is increased by the presence of any ammonium salts or carbon dioxide.

USES

It is used as an antacid.

As a calcium supplement in calcium deficiency.

Also used as food additive and homeopathic medicines.

As an inert filler for tablets and other pharmaceutical products.

As a source of dietary calcium supplement.

In the preparation of toothpaste.

Externally it is used as dentrifices because having abrasive property.

As a major component of blackboard chalk.

RESULT

Calcium carbonate was prepared and submitted.

EXPERIMENT NO -2

OBJECT: To prepare and submit magnesium carbonate (MgCO_3) .

REFERENCE

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 23-24.
2. Chatwal G.R, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 164-165.

REQUIRMENTS

Chemical required: Magnesium sulphate(MgSO_4), Sodium carbonate (Na_2CO_3)

Apparatus required: - Beaker, glass rod, measuring cylinder, funnel.

THEORY

Synonyms: Epsom salt

Chemical formula: MgCO_3

Molecular weight: 84.31

Magnesium carbonate is hydrated basic magnesium carbonate containing the equivalent of 40-50% of magnesium oxide. It occurs in two form, light and heavy magnesium carbonate. Dosage form is in form of oral suspension, tablets (200mg). Heavy Magnesium carbonate is different from light magnesium carbonate in density. 15g of heavy magnesium carbonate occupies a volume of 30 mL while light Magnesium carbonate occupies a volume of 125 mL. It is obtained by the double decomposition from magnesium sulphate and sodium carbonate.



PROCEDURE

Take 2.5g of Magnesium sulphate and 2.1g of sodium carbonate and dissolved separately in distilled water in 100mL beaker.

When both are completely dissolved, the solution are mixed and concentrated.

The residue is digested with boiling water for 30 minutes with water and the insoluble magnesium carbonate is filtered on calico cloth, washed until it becomes free from sulphate ions and dried in an oven.

PROPERTIES

It is a white granular powder

It is odourless and tasteless

It is insoluble in water and alcohol

It is soluble in dilute acids with effervescence.

USES

Magnesium carbonate used as an antacid and a laxative.

It is used as source of magnesium

It is used as anti-caking agent in cooking salt.

It is used as carbonate sources in soft drink.

In dentistry, magnesium carbonate is used as gypsum impression from which dental plates are made.

RESULT

Magnesium carbonate was prepared and submitted.

EXPERIMENT NO -3

OBJECT: To prepare and submit Zinc sulphate ($ZnSO_4$)

REFERENCE

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 51-52
2. Chatwal G.R, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 227-228

REQUIREMENTS

Chemical required: Zinc metal, dilute Sulphuric acid.

Apparatus required: - Beaker, funnel, glass rod & Measuring cylinder.

THEORY

Synonyms: White Vitrol

Chemical formula- $ZnSO_4 \cdot 7H_2O$

Molecular weight- 287.6

Zinc sulphate is widely used as an astringent. Astringents are the compound which brings about protein precipitation. The protein precipitation action of an astringent is due to presence of metallic ion having large charge. The metal would form complex with various polar groups present on the protein. In general astringents perform following function .

Stop the bleeding by coagulation of blood and constrict the small blood capillaries

Antiperspirant action by decreasing secretion of perspiration by reducing pore size of the skin

Anti-inflammatory action by decreasing supply of blood to the tissue.

Antimicrobial action by protein precipitation mechanism.

Zinc sulphate is prepared by digesting metallic zinc granules in dilute sulphuric acid



PROCEDURE

Take 5g of metallic zinc and add 50 mL of dilute sulphuric acid into a 250mL beaker.

Excess of zinc is added to remove excess of sulphuric acid.

After the effervescence ceases, the liquid is filtered concentrated and cooled.

Crystals of zinc sulphate are formed and separated by filtration at room temperature.

PROPERTIES

It is colorless transparent prism or small needle shape crystal or crystalline powder.

It is odorless but its taste is metallic and astringent.

It is very soluble in water and glycerin but insoluble in alcohol.

USES

Zinc sulphate is used as an astringent and applied externally.

It is used in the preparation of lotion.

It is used as a component of cosmetics and an ingredient of deodorant.

It is used as a preservative for woods.

Used as an anti-microbial treatment for sewage and as a herbicide.

Used as an emetic and as a dental impression material.

They are also used to treat diarrhoea.

RESULT

Zinc sulphate was prepared and submitted.

EXPERIMENT NO - 4

OBJECT: To prepare and submit Potash alum ($K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24 H_2O$)

REFERENCES

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 59-60
2. Chatwal G.R, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 223-224.

REQUIREMENTS

Chemical required: - Potassium sulphate, Aluminium sulphate.

Apparatus required: - Beaker, funnel, glass rod & Measuring cylinder.

THEORY

Synonyms: Alum

Chemical formula: ($K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24 H_2O$)

Molecular weight: 948.77

Alums are sulphates of a univalent metal and a trivalent metal.

Alum is also used as an astringent. Astringents are the compound which brings about protein precipitation. The protein precipitation action of an astringent is due to presence of metallic ion having large charge. The metal would form complex with various polar groups present on the protein. In general astringents perform following function .

Stop the bleeding by coagulation of blood and constrict the small blood capillaries

Antiperspirant action by decreasing secretion of perspiration by reducing pore size of the skin

Anti-inflammatory action by decreasing supply of blood to the tissue.

Antimicrobial action by protein precipitation mechanism.

It is prepared by adding a concentrated solution of potassium sulphate to a hot solution of aluminium sulphate.



PROCEDURE

Take 6.3 g of potassium sulphate (K_2SO_4) and dissolved in 10 mL of dissolved water & 2.5 g of Aluminium sulphate $\text{Al}_2(\text{SO}_4)_3$, is dissolved in water separately.

Heat both the solution and Mix slowly with continous stirring.

It is cooled in ice bath for half an hour.

Crystals of potash alum separates out, generally crystals are octahedral in shape.

Filter the solution and dry alum.

PROPERTIES

Alum occurs as large colorless crystal.

It is odorless with a sweet astringent taste.

Its solution is acidic to litmus.

Alum are freely soluble in water but slowly dissolve in glycerin and in soluble in alcohol

USES

Alum is used as aftershave due to its astringent property. It can be rubbed on freshly shaved face.

Used in the preparation of cosmetics like deodorant

Alum powder is used to reduce and prevent bleeding due to small cut.

Used as a mouthwash & tooth powder.

Used as a mordant in dying industry.

Used as protein precipitant.

Used in fire extinguisher due to its flame retardant property.

RESULT

Potash alum was prepared and submitted.

EXPERIMENT NO -5

OBJECT: To prepare and submit Boric acid.

REFERENCE

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 23-24.
2. Chatwal G.R, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 127-128.

REQUIREMENTS

Chemical required: Borax ($\text{Na}_2\text{B}_4\text{O}_7$), Dilute sulphuric acid (H_2SO_4)

Apparatus required: Beaker, Pipette, Measuring cylinder, glass rod, funnel.

THEORY

Synonyms: Boro flax, Borocic acid

Chemical formula: H_3BO_3

Molecular weight: 61.83

Boric acid is a weak acid. It is a local anti-infective agent possessing weak bacterostatic and fungistatic properties. Anti-infective agent are the compound which reduce or prevent infection from the microbes like bacteria, fungi and protozoa.



PROCEDURE

Take 5 g of borax in a beaker and dissolved in 10mL of water.

Add 40 mL of dilute sulphuric acid, H_2SO_4 with continuous stirring and boiled the solution.

Aqueous, solution is then cooled in ice baths for 45 minutes.

White crystals are obtained and dried between filter paper.

Collect the product from spatula.

PROPERTIES

Boric acid is solid crystalline powder.

It is colorless, odorless with slightly acidic and bitter in taste and unctuous in touch.

It is soluble in water and alcohol.

It is a weak acid.

It is stable in air.

USES

Boric acid is used as an insecticide for controlling house holding pests like termites, ants, and small insects.

It is used as an antiseptic for treating minor burn and cuts

Solution of boric acid is used to wash eyes in conjunctivitis.

It is used as an astringent, an antiseptic and as a antimicrobial agent

Used in the preparation of eye-drops.

Used as wood preservative.

Used in the preparation mouth wash and skin lotion.

Boric acid is used in heat resistant borosilicate and other heat resistant glassware.

RESULT:

Boric acid was prepared and submitted.

EXPERIMENT NO -6

OBJECT: To prepare and submit aluminum hydroxide.

REFERENCES

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 45-46
2. Chatwal GR, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 156-157

REQUIREMENTS

Chemical required: Aluminium chloride (AlCl₃), Ammonium hydroxide (NH₄OH), Sodium carbonate (Na₂CO₃)

Apparatus required: Measuring cylinder, beaker, glass rod, and funnel.

THEORY

Synonyms: Hydrated Aluminium oxide

Chemical formula: Al(OH)₃

Molecular weight: 78

Aluminium hydroxide Al(OH)₃ gel is an aqueous suspension of hydrated aluminium oxide with different amounts of basic aluminium carbonate & bicarbonate. It is a white, light, odourless, tasteless, amorphous powder, insoluble in water & alcohol. It acts as an amphoteric substance. It behaves as an antacid hence raise the pH of solution.



PROCEUERE

Weigh 6 g of Aluminium chloride & 18 g of Sodium carbonate & mix it.

Add 30mL distilled water.

Gradually add NH_4OH until white gel is formed. Cool in ice bath, white ppt is formed.

AlCl_3 is hydrolyzed by NH_4OH and form $\text{Al}(\text{OH})_3$ gel.

USES

Used as an Antacid.

It has astringent properties.

RESULT

Aluminium hydroxide was prepared and submitted.

EXPERIMENT NO-7

OBJECT: - To perform limit test for chloride in given sample.

REFERENCE

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 56.

REQUIREMENTS: -

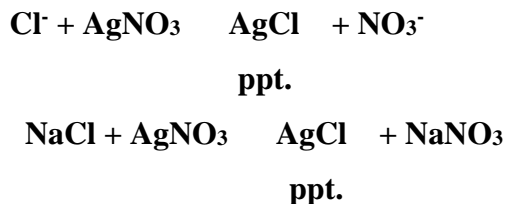
Chemical required: Silver nitrate, dilute nitric acid, sodium chloride etc.

Apparatus required: - Measuring cylinder, glass rod and Nessler's cylinder.

THEORY

Limit test are quantitative or semi quantitative test designed to identify and control small quantity of impurities which are likely to be present in the substance.

This test involve the reaction of silver nitrate with soluble chloride to form the ppt. of silver chloride which is insoluble in dilute HNO₃. The extent of the precipitation depends upon the amount of silver chloride formed i.e. on the amount of chloride ions present in the substance. The opalescence produced in test solution is compared with a reference/standard solution under the same experimental conditions.



PROCEDURE

STANDARD

1mL of 0.05845% w/v solution of NaCl is taken in Nessler's cylinder

Add 10 mL of Dil. HNO₃

Makeup the volume upto 50 mL with distilled water.

Now add 1mL of silver nitrate to this solution.

Stirrer the solution with glass rod and allow to stand for 5 minutes.

TEST

Dissolve specified quantity of substances as per I.P. monograph in 10mL of distilled water.

Add 10 mL of Dil. Nitric acid.

Makeup the volume upto 50 mL with distilled water.

Now add 1mL of silver nitrate to this solution.

Stirrer the solution with glass rod and allow to stand for 5 minutes.

OBSERVATION

The opalescence of test solution is less/more than standard solution.

If opalescence of test solution has been less than the standard opalescence, the sample will pass the limit test.

RESULT

Limit test for chloride was performed

EXPERIMENT NO -8

OBJECT: To perform limit test of Sulphate in given sample.

REFERENCES

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 57.

REQUIREMENTS

Chemical required: Hydrochloric acid, Barium sulphate and Barium chloride

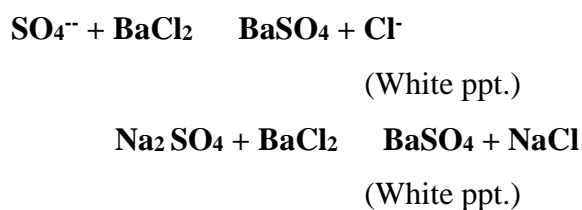
Apparatus required: Measuring cylinder, glass rod, pipette and Nessler's cylinder etc.

THEORY

Limit test are quantitative or semi quantitative test designed to identify and control small quantity of impurities which are likely to be present in the substance

This test involves the reaction of Barium chloride with soluble sulphate to form the precipitate of Barium sulphate which is insoluble in dilute hydrochloric acid. The Barium sulphate precipitate is white in colour.

REACTION



PROCEDURE

STANDARD

Take 1mL of 0.1089 w/v of Na₂SO₄ or K₂SO₄ as per I.P. in nessler cylinder.

Add 2 mL of dilute Hydrochloric acid.

Makeup the volume upto 45 mL with distilled water.

Add in this solution of 5 mL of Barium sulphate reagent

Stirrer the solution with glass rod and allow to stand for 5 minutes.

TEST

Dissolve the specific quantity of test substances in 10 mL of distilled water.

Add 2 mL of dilute Hydrochloric acid.

Makeup the volume upto 45 mL with distilled water.

Add in this solution of 5 mL of Barium sulphate reagent

Stirrer the solution with glass rod and allow to stand for 5 minutes.

OBSERVATION

The opalescence of test solution is less/more than standard solution.

If opalescence of test solution has been less than the standard opalescence, the sample will pass the limit test.

RESULT

Limit test for sulphate was performed

EXPERIMENT NO -9

OBJECT: To perform limit test of iron in given sample.

REFERENCE:-

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 58.

REQUIREMENTS

Chemical required: Thioglycolic acid, citric acid, Ammonia solution, ferric ammonium sulphate.

Apparatus required: Measuring cylinder, glass rod, pipette and Nessler's cylinder.

THEORY

Limit test are quantitative or semi quantitative test designed to identify and control small quantity of impurities which are likely to be present in the substance.

The limit test of iron is based on the reaction between iron and thioglycolic acid in the presence of citric acid in an ammoniacal solution. Citric acid prevents precipitation of iron with Ammonia. A deep reddish purple colour is formed.

Ferrous thioglycolate is colourless in acidic medium but in alkaline medium it gives purple colour.



PROCEDURE

STANDARD

Take standard 1.5 mL of Iron solution.

Add 1.5 mL of iron free citric acid to this solution and 1.5 mL of thioglycolic acid and make the solution alkaline.

Volume make up to 50 mL.

Stirir the solution allow to stand for 5 minutes.

TEST

Dissolve specific quantity of substances being examined dissolved in water.

Add 1.5 mL of iron free citric acid to this solution and 1.5 mL of thioglycollic acid and make the solution alkaline.

Volume make up to 50 mL.

Stirir the solution allow to stand for 5 minutes

RESULT

Limit test for Iron was performed.

EXPERIMENT NO -10

OBJECT: To perform the identification test of boric acid in given sample.

REFERENCE

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 23-24

REQUIREMENTS:-

Chemical required: Boric acid, sulphuric acid, Borax.

Apparatus required: Pipette, beaker, conical flask, volumetric flask, measuring cylinder.

THEORY

Boric acid is a weak acid. It is a local anti-infective agent possessing weak bacterostatic and fungistatic properties. It is obtained from Borax. It occurs as colorless plates or white crystalline or white crystalline powder which is greasy in touch. It is odourless and taste is slightly acidic and bitter. It is soluble in water and in alcohol, freely soluble in boiling water, in boiling alcohol and in glycerine. Identification test of boric acid is based on flame test.



PROCEDURE:-

0.5 g of substance being examined is taken in a test tube and add 5mL of methanol to it. The mixture is ignited. The alcohol burn with green edge flame. Colour is due to the formation of methyl borate or ethyl borate.

3 g of substance being examined is dissolve in 90mL of distilled water and the mixture is cool.

Now check the acidity of the solution with litmus paper.

The solution is faintly acidic.

RESULT

Identification test of boric acid was performed.

EXPERIMENT NO -11

OBJECT: To perform the identification test of ammonium chloride.

REFERENCE

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 19-20.
2. Chatwal GR, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 256-257

REQUIREMENTS

Chemical required: Ammonium chloride, nitric acid, Nitrobenzen, Silver nitrate and Ferric ammonium sulphate.

Apparatus required: Volumetric flask, conical flask, Burette, Pipette, Glass rod.

THEORY

Ammonium chloride may be assayed by the precipitation titration technique using Volhard's Method. As ammonium chloride is a salt of a weak base acid, its aqueous solution is acidic. Due to this, the reaction with silver nitrate by direct titration is not able to complete the process; hence, Volhard's Method (back titration method) is used.



PROCEDURE

Take 0.2g of ammonium chloride add small amount of nitric acid and silver nitrate. A white precipitate of silver chloride is formed. When dilute ammonia solution is added to the precipitate, it dissolved due to the formation of complex.

RESULT:

Identification test of ammonium chloride was performed.

EXPERIMENT NO -12

OBJECT: - To identify cation & anion in given pharmaceutical compounds.

REFERENCE:-

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed 1st, 2008, pp 19-25
2. Chatwal GR, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 437-439

OBSERVATION

S.NO.	IONS	TEST	OBSERVATION	INFERENCE
1.	Acetate	a) original solution + dil. H ₂ SO ₄ b) original solution + FeCl ₃	Smell of vinegar Radish brown ppt	Acetate may be present. Acetate confirmed.
2.	Chloride	a) Salt + conc. H ₂ SO ₄ take a rod dipped in NH ₃ solution to mouth of test tube. b) salt + water + dil. HNO ₃ + AgNO ₃	Pungent smell, white fumes of NH ₄ Cl obtained Curdy white ppt	Cl ⁻ may be present. Cl ⁻ confirmed.
3.	Iodide	a) salt + water + dil. HNO ₃ + AgNO ₃	Yellow colour	I ⁻ present

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		b) salt + conc H ₂ SO ₄	Violet fumes obtain	I ⁻ present
4.	Bromide	a) salt + conc.H ₂ SO ₄ b) original solution + Cl ⁻ solution + 2-3 drop of CHCl ₃	Reddish brown fumes obtained which intensity on adding MnO ₂ Reddish color.	Br ⁻ may be present. Br ⁻ confirmed.
5	Carbonate	a) salt + dil. H ₂ SO ₄ if CO ₂ is passed to lime water, it turns milky b) original solution + MgSO ₄	Effervescence obtained due to liberation of CO ₂ White ppt obtained	CO ₃ ²⁻ may be present CO ₃ ²⁻ confirmed.

6.	Sulphate	a) substances + BaCl ₂ b) Original solution + lead acetate.	White ppt White ppt	SO ₄ ²⁻ may be present. SO ₄ ²⁻ confirmed.
7.	Nitrate	a) Original solution + conc. H ₂ SO ₄ + FeSO ₄ during heating add copper chips. b) Original solution + FeSO ₄ gradually add conc. H ₂ SO ₄	Reddish brown fumes Brown ring is formed at the junction of 2 layer.	NO ₃ ⁻ may be present. NO ₃ ⁻ confirmed.
8.	Bicarbonate	a) Original solution + MgSO ₄	No ppt then boil white ppt formed	HCO ₃ ⁻ Present

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9.	Ba ⁺⁺	a) salt + Dil. HCl + Dil.H ₂ SO ₄	White ppt obtain insoluble in HNO ₃ .	Ba ⁺⁺ present
10.	Fe ⁺³	a) Original sol ⁿ + Pottasium ferrocynide b) Original sol ⁿ + HCl + NH ₄ SCN	Intense blue colour obtained Blood red colour obtain	Fe ⁺³ may be present Fe ⁺³ confirmed.
11.	Fe ⁺²	a) Original sol ⁿ + Dil. H ₂ SO ₄ + Phenanthraline Salt + Pottasium ferrocynide	Intense red colour obtained White ppt form which rapidly becomes blue	Fe ⁺² present Fe ⁺² present
12.	Lead	a) Original sol ⁿ + dil. Acetic acid + Pottasium chromate b) Original sol ⁿ + dil. Acetic acid + KI	Yellow ppt obtained Yellow ppt obtained	Lead present Lead present
13.	Zinc	a) Original sol ⁿ + NaOH b) Original sol ⁿ + Potassium ferrocynide	White ppt obtained White ppt obtained	Zn ⁺² Present Zn ⁺² Present