

S.A.RAJA PHARMACY COLLEGE

VADAKANGULAM- 627 116

TIRUNELVELI DISTRICT

SUBJECT: PHARMACEUTICAL IN ORGANIC CHEMISTRY



PRACTICAL MANUAL BOOK

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LIMIT TEST

INTRODUCTION

Limit test are quantitative or semi - quantitative tests which are designed to detect and control small quantities of impurities which are likely to be present in the substance.

The tests require a standard containing a definite amount of impurity to be set up at the same time and under the same conditions of the experiment. In this way, it is possible to compare the amount of the impurity in the substance with a standard of known concentration and find out whether the impurity is within or excess of the limit prescribed.

For this purpose those tests make use of simple comparisons of opalescence, turbidity or colour with standard as prescribed in the pharmacopoeias. By taking different quantities of the test substance it is possible to vary the limits of the impurities permitted for each substance. No numerical values are given for the permitted limits because other impurities also may interfere with the tests. Variations in time and method of performing the test also influence the tests.

Ex No: 1

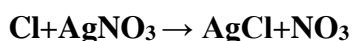
Date: LIMIT TEST FOR CHLORIDE I. P. 1985

AIM:

To perform the limit test for chlorides on the given sample as per IP.

PRINCIPLE:

The principle involved in the limit test for chlorides is based on the well-known reaction between silver nitrate and soluble chlorides forming a precipitate of silver chloride which is insoluble in dilute nitric acid.



The test solution becomes turbidity the extend of turbidity depends upon the amount of silver chlorides produced. This in turns depends upon the amount of chloride present in the test solution. The opalescence of standard containing known quantity of chloride. If the turbidity found in the sample is less than the standard. The sample limit test for chloride is passed. It must be viewed transversely against the dark background.

PROCEDURE:

Tate two 50ml Nessler's cylinders label one as test and the other as standard.

S. No.	Test	Standard
1	Dissolve the specify quantity of the substance (1 gram of dextrose) in distilled water or prepare a solution as directed in the test in the nessler's cylinder	Pipette out 1 ml of 0.05845% w/v solution of sodium chloride into the nessler's cylinder.
2	Add 10 ml of dilute nitric acid.	Add 10 ml of dilute nitric acid.
3	Dilute to 50 ml with water.	Dilute to 50 ml with water.
4	Add 1 ml of silver nitrate solution.	Add 1 ml of silver nitrate solution.
5	Stir immediately with a glass rod and allowed to stand for 5 minutes.	Stir immediately with a glass rod and allowed to stand for 5 minutes.

OBSERVATION:

The opalescence produced of the test solution is ----- than the standard solution.

REPORT:

The given unknown sample ----- the limit test for chloride.

Ex No: 2

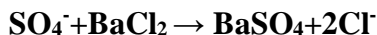
Date: **LIMIT TEST FOR SULPHATES I. P.1985**

AIM:

To perform the limit test for sulphates for the given sample.

PRINCIPLE:

This is based on the reaction between the barium chlorides and soluble sulphates in the presence of dilute hydrochloric acid.



The test solution becomes turbid the extent of turbidity depends on the amount of barium sulphate produced. Which in turn depends upon the amount of test solution. The substance passes the limit test if the turbidity produced in the sample is less than the standard. If the turbidity is more than the standard, it fails the test. The barium sulphate reagent contains mainly barium chloride, sulphate free alcohol and small amount of potassium sulphate. Alcohols prevent the supersaturation and more uniform turbidity is formed. The addition of potassium sulphate increases the sensitivity of test. The barium chloride present in the barium sulphate reagent in a small quantity acts as a seeding agent for the precipitation of barium sulphate. If sulphate present in the test sample.

PROCEDURE:

Take two 50 ml Nessler cylinder label one as test and the other as standard.

Test	Standard
<ul style="list-style-type: none">Dissolve the specified quantity of the substance in water and transfer to a 50 ml nessler's cylinder.	<ul style="list-style-type: none">Pipette out 1 ml of 0.1089 percent w/v solution of potassium sulphate into the nessler's cylinder.
<ul style="list-style-type: none">Add 2ml of dilute hydrochloric acid.	<ul style="list-style-type: none">Add 2 ml of dilute hydrochloric acid.
<ul style="list-style-type: none">Dilute to 45 ml with water.	<ul style="list-style-type: none">Dilute to 45 ml with water.
<ul style="list-style-type: none">Add 5 ml of barium sulphate reagent.	<ul style="list-style-type: none">Add 5 ml of barium sulphate reagent.
<ul style="list-style-type: none">Stir immediately with a glass rod and allow to stand for 5 minutes.	<ul style="list-style-type: none">Stir immediately with a glass rod and allowed to stand for 5 minutes.

OBSERVATION:

The opalescence of the test solution is ----- than the standard solution

REPORT:

The given sample ----- the limit test for sulphate.

Ex No: 3

Date:

LIMIT TEST FOR IRON I.P. 1985

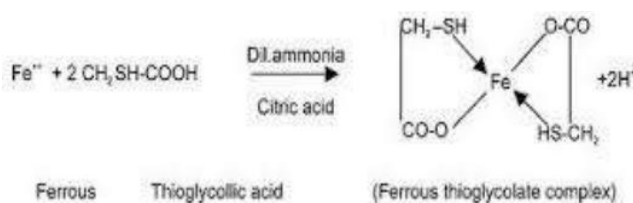
AIM:

To perform the limit test for iron on the given sample

PRINCIPLE:

This test depends upon the reaction of iron with thioglycollic acid in the presence of citric acid and ammonia when a pale pink to deep reddish purple colour is produced. Ferric iron is reduced to ferrous iron by the thioglycollic acid and the compound produced is ferrous thioglycollate. This is stable in the absence of air and fades when exposed to air due to oxidation. The original state of oxidation as thioglycollic acid is a reducing agent and reduced ferric ion to ferrous.

The colour produced is due to the reaction between ferrous compound and thioglycollic acid which forms ferrous thioglycollate. This is coloured in alkaline solution and colourless in acid as neutral solution.



PURPOSE OF ADDING AMMONIA

Ammonia is added to make the solution alkaline in which the solution ammonia forms ferrous thioglycollate it gives colour.

PURPOSE OF ADDING CITRIC ACID

Citric acid prevents the precipitation of iron by ammonia as ferrous hydroxide.

PROCEDURE:

Take two 50 ml Nessler cylinders. Label one as “Test” and the other as “Standard”.

Test	Standard
<ul style="list-style-type: none">Dissolve the specified quantity of the substance (1g of sodium Chloride) in 40ml of water or use 10 ml of the solution as prescribed in the monograph and transfer to a nessler cylinder.	<ul style="list-style-type: none">Dilute 2ml of standard iron solution with 40ml of water in a nessler cylinder.
<ul style="list-style-type: none">Add 2ml of 20% W/v solution of iron free citric acid and 0.1ml of thioglycolic and mix.	<ul style="list-style-type: none">Add 2 ml of 20% W/v of solution of iron free citric acid and 0.1ml of thioglycolic and mix.
<ul style="list-style-type: none">Make alkaline with iron free ammonia solution.	<ul style="list-style-type: none">Make alkaline with iron free ammonia solution.
<ul style="list-style-type: none">Dilute to 50ml with water.	<ul style="list-style-type: none">Dilute to 50ml with water
<ul style="list-style-type: none">Allow to stand for 5 minutes.	<ul style="list-style-type: none">Allow to stand for 5 minutes.

OBSERVATION:

The intensity of the test solution is ----- than the standard solution.

REPORT:

The given sample ----- the limit test for iron.

Date:

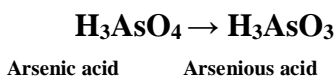
LIMIT TEST FOR ARSENIC I. P. 1985

AIM:

To perform the limit test for arsenic acid on the given sample

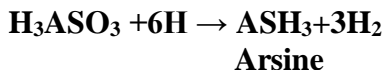
PRINCIPLE:

The test substance is dissolved in hydrochloric acid or an aqueous solution or extract is acidified. Some substances have to be specially treated for making a solution suitable for the test. The arsenic present in the sample is converted to either arsenious acid or arsenic acid depending on its valency state. Then it is further treated with a reducing agent such as stannous chloride. Arsenic acid is reduced to arsenious acid. In the I.P. stannated hydrochloric acid (stannous chloride mixed with hydrochloric acid) is added to the substance.

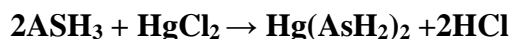


Potassium iodide which is also added forms hydrochloric acid which also reduces arsenic acid to arsenious acid.

The arsenious acid is further reduced to arsine by nascent hydrogen produced by the action of granulated zinc and hydrochloric acid.



When arsine comes into contact with dry paper saturated with mercuric Chloride, it produces a yellow or brown stain.



The intensity of the stain is compared by daylight with a standard stain which is similar and simultaneously prepared by taking a specified quantity of dilute arsenic solution in place of the test substance. If the test stain is less in intensity of colour than the standard stain, the sample passes the test.

All the reagents used excepting strong and dilute arsenic solutions should be arsenic-free and are designated as AsT. A standard stain prepared by taking 1 ml of dilute arsenic solution, AsT and compared with the test stain produced limit of arsenic is 1 part per million.

APPARATUS:

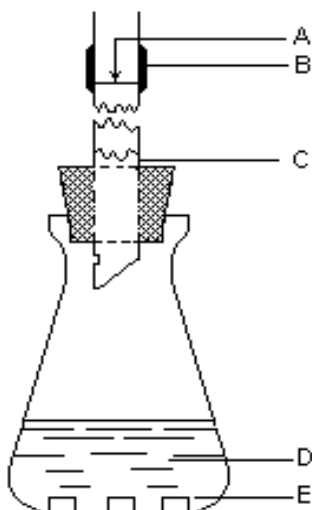
The apparatus consists of a wide mouthed glass bottle (about 100 ml capacity) or conical flask fitted with a rubber bung or ground glass stopper. A glass tube of specified dimensions is passed through the rubber bung.

The internal diameter (5mm) is important and should be uniform throughout the tube is open at the lower end. Near the lower end a small hole is present to allow condensed moisture to escape.

The tube is packed tightly with cotton wool saturated with lead acetate solution and dried. This is to drop any hydrogen sulphide which may be produced during the reaction if any sulphur impurity is present in the substance.

The mercuric chloride paper is fixed at the upper end of the tube between two rubber bungs by means of a spring clip. The two rubber bungs contain the tube into two parts and the mercuric chloride paper proper is correctly positioned between them.

APPARATUS FOR LIMIT TEST FOR ARSENIC



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

PROCEDURE:

Take two 120 ml wide- mouthed bottles or conical flasks with the attachments and label one as “Test” and the other as “standard”.

Test	Standard
• Weigh accurately 10g of the sample and dissolve in 50 ml of water. Transfer to the bottle.	• Take in the bottle accurately 1 ml of dilute arsenic solution. Add 50ml of water.
• Add 10 ml of stannated hydrochloric acid AsT.	• Add 10 ml of stannated hydrochloric acid AsT.
• Add 1g of potassium iodide AsT and 10 g of zinc AsT.	• Add 1g of potassium iodide AsT and 10 g of zinc AsT.
• Place the cork immediately over the bottle with the attachments and immerse the bottle in a water bath at suitable temperature.	• Place the cork immediately over the bottle with the attachments and immerse the bottle in a water bath at suitable temperature.
• Allow the reaction to go on in the cold for forty minutes.	• Allow the the reaction to go on in the cold for forty minutes.
• Remove the mercuric chloride paper at the end of forty minutes.	• Remove the mercuric chloride paper at the end of forty minutes.

OBSERVATION:

The intensity of yellow stain produced in the sample ----- than the standard stain.

REPORT:

The given sample ----- the limit test for arsenic.

Ex No: 5

Date:

MODIFIED PROCEDURE FOR CERTAIN LIMIT TEST

AIM:

To perform the limit test for chloride and sulphate in potassium permanganate.

PRINCIPLE:

The potassium permanganate is highly coloured substances. If this limit test done in the usual way it will be difficult to make observation. So potassium permanganate is eliminate by reduction with alcohol. This can be called pretreatment.

The sample is dissolve in water and heated on water bath; alcohol is added it is filtered to remove the precipitate manganese dioxide. The filterate is colourless and can be used for performing limit test for chloride and sulphate in usual way.



PROCEDURE:

- Dissolve 1.5 gm of sample in a 50 ml of distilled water.
- Heat on water bath and add gradually 6 ml of ethanol 95%.
- Cool, then dilute to 60 ml with distilled wster and filter. The filterate (solution A) is colourless.

FOR LIMIT TEST FOR CHLORIDE

Take 40 ml of solution A and do the limit tests for chloride.

FOR LIMIT TEST FOR SULPHATE

Take 10 ml of solution A and do the limit tests for sulphate.

OBSERVATION

The opalescence of the test solution is -----than the standard solution.

REPORT

The limit test for chloride and sulphate is -----.

Ex No: 6

Date:

PREPARATION OF BORIC ACID

AIM

To prepare and submit Boric acid from borax and report its practical yield.

PRINCIPLE:

Boric acid is a weak acid; it is prepared from borax. The borax is reacted with concentrated sulphuric acid in the presence of water to give a crude compound of boric acid.

CHEMICAL REACTION:



INGREDIENTS REQUIRED:-

Borax – 20gm

Conc. Sulphuric acid – 10 ml

PROCEDURE:-

Dissolve 20 gm of borax in 100 ml water and heat it as necessary to dissolve the borax. Add to the solution about 10 ml of conc. Sulphuric acid slowly with constant stirring. Cool and filter it. Wash the precipitate which is to remove sulphate dry product and weigh it.

MEDICINAL USE:-

Anti infective

REPORT:-

The Boric acid was prepared and submitted.

The practical yield was found to be -----gm.

Ex No: 7

Date:

PREPARATION OF CALCIUM CARBONATE

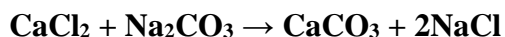
AIM

To prepare and submit Calcium carbonate from calcium chloride and report its practical yield.

PRINCIPLE:

Calcium carbonate can be prepared by mixing boiling solutions of calcium chloride and sodium carbonate. The precipitated calcium carbonate is filtered, washed free from chloride and dried.

CHEMICAL REACTION:



INGREDIENTS REQUIRED:-

Calcium chloride – 22gm

Anhydrous sodium carbonate – 17gm

PROCEDURE:-

Dissolve about 22 gm of anhydrous calcium chloride in boiling water. Dissolve about 17 gms of anhydrous sodium carbonate in boiling water. Add this sodium carbonate solution into the calcium chloride solution and mix well. Cool and filter in a caligo filter wash the precipitate on the filter with distilled water till the filtrate is free from chloride. Dry the precipitate at 105⁰C' in a hot air oven.

MEDICINAL USE:-

Calcium replenshier

REPORT:-

- i) The Calcium carbonate was prepared and submitted.
- ii) The practical yield was found to be -----gm.

Ex No: 8

Date:

PREPARATION OF MAGNESIUM SULPHATE

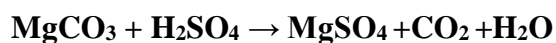
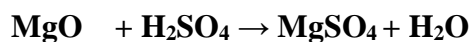
AIM

To prepare and submit Magnesium sulphate and report its practical yield.

PRINCIPLE:

Magnesium sulphate is prepared by dissolving either magnesium oxide or magnesium carbonate in diluted sulphuric acid. After evaporating the solution, the anhydrous salt is obtained.

CHEMICAL REACTION:



INGREDIENTS REQUIRED:-

Magnesium oxide – 8gm

Conc. Sulphuric acid – 20 ml

PROCEDURE:-

Take 8 gm of magnesium oxide in a 100 ml beaker. Separately add 20 ml conc. Sulphuric acid to about 100 ml of water. Add magnesium oxide gradually and with stirring to the diluted acid. And evaporate the solution concentrated to about ¼ of its volume and cool. Filter the magnesium sulphate precipitated out and dried it.

MEDICINAL USE:-

Calcium replenshier

REPORT:-

- i) The Magnesium sulphate was prepared and submitted
- ii) The practical yield was found to be -----gm.

Ex No: 9

Date:

PREPARATION OF POTASH ALUM

AIM

To prepare and submit potash alum and report its practical yield.

PRINCIPLE:

Potash alum is a sulphate salt of potassium and aluminium in the hydrated form. In general, alum has the general formula $X_2SO_4 \cdot Y_2(SO_4)_3 \cdot 24H_2O$ where x and y represent mono and trivalent metal cation respectively. Potash alum is prepared by mixing solutions of potassium sulphate and aluminium sulphate in water.

CHEMICAL REACTION:



INGREDIENTS REQUIRED:-

Potassium sulphate – 5.8gm

Aluminium sulphate – 11.4 gm

Water – Q.S

PROCEDURE:-

Dissolve separately the required quantity of potassium sulphate and aluminium sulphate in minimum water. Warm the solutions if necessary. The homogeneous solutions are mixed and the resulting solution is left undisturbed overnight during which clear transparent crystals are obtained. These were washed, recrystallised, dried and submitted.

MEDICINAL USE:-

Astringent

REPORT:-

- i) The Potash alum was prepared and submitted.
- ii) The practical yield was found to be -----gm.

Ex No: 10

Date:

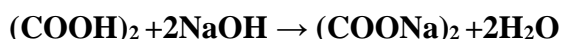
STANDARDIZATION OF N/10 SODIUM HYDROXIDE

AIM:

To find out the normality of the given N /10 sodium hydroxide solution.

PRINCIPLE:

Sodium hydroxide is a strong base. So it can be titrated with weak acid like oxalic acid. Phenolphthalein is used as an indicator. End point is the appearance of pale pink colour.



Oxalic acid **sodium oxalate**

PROCEDURE:

Weigh about 0.63 g of oxalic acid is transfer into a 100 ml standard flask it is dissolved using distilled water and make up to the mark and from this 20 ml is pipette out into a clean conical flask. Add two drops of phenolphthalein indicator solution. Titrate with N/10 sodium hydroxide solution which is taken in the burette. End point is the appearance of permanent pale pink colour. The titration is repeated to get concordant value.

CALCULATION:

Normality of oxalic acid = weight taken/weight to be taken

Oxalic acid vs Sodium hydroxide

S. NO	Volume of oxalic acid	Burette Reading		Volume of N/10 NaOH	Indicator	End Point
		Initial (ml)	Final (ml)			

Strength of sodium hydroxide

$$V_1N_1 = V_2N_2$$

REPORT:

The Normality of the given N/10 Sodium Hydroxide = -----N/10.

Ex No: 11

Date:

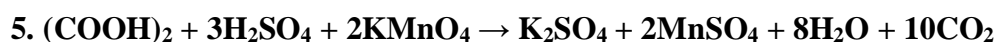
STANDARDATION OF N / 10 POTASIUM PERMANGANTAE

AIM:

To Standardize the approximately N / 10 potassium permanganate solution.

PRINCIPLE:

Potassium permanganate solution is standardized by titration against standard oxalic acid solution. Oxalic acid in the presence of dilute sulphuric acid is oxidized by potassium permanganate to carbon dioxide and water. The oxalic acid solution should be warmed to 70°C before the titration, as otherwise the reaction is slow.



Oxalic acid

PROCEDURE:

Weight accurately about 0.63 g of oxalic acid, and transfer to a 100ml standard flask. Add enough water to dissolve and make up to the mark. Stopper the flask and shake well.

Pipette out 20ml of the standard oxalic acid solution into a clean conical flask and add 20ml of dilute sulphuric acid. Warm this solution to 70° C and titrate with potassium permanganate till a permanent faint pink colour appears. Repeat the titration to get concordant values.

CALCULATION:

Normality of oxalic acid = = weight taken/weight to be taken

Oxalic acid vs Potassium permanganate

S. NO	Volume of N/10 oxalic acid	Burette Reading		Volume of N/10 KmnO ₄	Indicator	End Point
		Initial (ml)	Final (ml)			

Strength of Potassium permanganate

$$V_1N_1 = V_2N_2$$

REPORT:

The normality of given N / 10 potassium permanganate solution = 0.9823 N / 10.

Ex No: 12

Date:

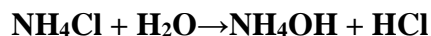
ASSAY OF AMMONIUM CHLORIDE

AIM:

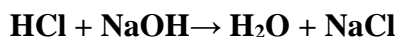
To determine the percentage purity of given sample.

PRINCIPLE:

Ammonium chloride is dissolved in water to form ammonium hydroxide and this react with formaldehyde to form hexamine. This is basically acid base titration where hydrochloric acid is treated against sodium formaldehyde solution may contain small amount of formic acid formed due to atmospheric oxidation. This is carefully neutralized before adding to the solution of ammonium chloride.



Hexamine



PROCEDURE:

Weigh accurately about 0.1 g of ammonium chloride and transfer to a clean conical flask. Dissolve in 20 ml of water. Add a mixture of 5 ml of formaldehyde, previously neutralised to phenolphthalein and 20ml of water. Allow to stand for 10 minutes. Then titrate slowly with sodium hydroxide solution using phenolphthalein solution as indicator until a permanent pale pink colour appears. Each ml of sodium hydroxide is equivalent to 0.005349 g of NH_4Cl solution.

Standardization of N/10 NaOH:

Weigh accurately about 0.63 g of oxalic acid and transfer into standard flask. Make up the volume to 100ml with distilled water pipette out 20 ml of this solution and add phenolphthalein as an indicator and titrate against N/10 NaOH. The end point is the appearance of permanent pale pink colour.

Standardization of N/10 NaOH:

Calculation:

Normality of oxalic acid = weight taken/weight to be taken

Oxalic acid vs Sodium hydroxide

S. NO	Volume of oxalic acid	Burette Reading		Volume of N/10 NaoH	Indicator	End Point
		Initial (ml)	Final (ml)			

Strength of sodium hydroxide

$$V_1N_1 = V_2N_2$$

Assay of Ammonium Chloride

Ammonium chloride vs Sodium hydroxide

S. NO	Content of flask	Burette Reading		Volume of N/10 NaoH(ml)	Indicator	End Point
		Intial(ml)	Final(ml)			

The amount of ammonium chloride = $\frac{\text{Titre value} \times \text{Normality of N/10NaOH} \times \text{Eq weight factor}}{\text{Weight taken}}$

% purity of ammonium chloride = amount present x 100

REPORT:

i)The amount of ammonium chloride present is the given sample = ----- w/w.

ii)The percentage purity of ammonium chloride =----- % w/w.

Ex No: 13

Date: **ASSAY OF FERROUS SULPHATE**

AIM:

To assay the given sample of ferrous sulphate and calculate its percentage purity (%w/w).

PRINICIPLE:

Ferrous sulphate solution is mixed with dilute sulphuric acid and titrated against standard potassium permanganate. Ferrous sulphate is oxidized to ferric sulphate by potassium permanganate which acts as a self –indicator.



PROCEDURE:

Weight accurately about 1 g of the sample of ferrous sulphate and dissolve in 20 ml of dilute sulphuric acid in a clean conical flask. Titrate with standard potassium permanganate till a permanent pale pink colour appears. Each ml of Potassium permanganate is equivalent to 0.0278 g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$.

STANDARDISATION OF N/10 POTASSIUM PERMANGANATE:

Weight accurately about 0.63 g of oxalic acid, and transfer to a 100ml standard flask. Add enough water to dissolve and make up to the mark. Stopper the flask and shake well. Pipette out 20ml of the standard oxalic acid solution into a clean conical flask and add 20ml of dilute sulphuric acid. Warm this solution to 70° C and titrate with potassium permanganate till a permanent faint pink colour appears.

Calculation:

Normality of oxalic acid = = weight taken/weight to be taken

Oxalic acid vs Potassium permanganate

S. NO	Volume of N/10 oxalic acid	Burette Reading		Volume of N/10 KmnO_4	Indicator	End Point
		Initial (ml)	Final (ml)			

Strength of Potassium permanganate

$$V_1N_1 = V_2N_2$$

Assay of Ferrous sulphate

Ferrous sulphate vs Sodium hydroxide

S. NO	Content of flask	Burette Reading		Volume of N/10 NaoH(ml)	Indicator	End Point
		Intial(ml)	Final(ml)			

The amount of Ferrous sulphate= $\frac{\text{Titre value} \times \text{Normality of N/10NaOH} \times \text{Eqt weight factor}}{\text{Weight taken}}$

% purity of Ferrous sulphate = amount present x 100

REPORT:

The percentage purity of the given ferrous sulphate = ----- % w/w.

Ex No: 14

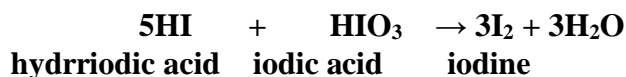
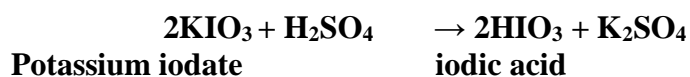
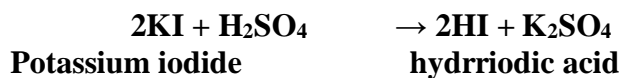
Date: TEST FOR PURITY OF IODATE IN POTASSIUM IODIDE

AIM:

To perform the test for purity in potassium iodide.

PRINCIPLE:

Iodate in potassium iodide is tested by adding dil. sulphuric acid and starch solution, to an aqueous solution of the sample no blue colour should be produced. Iodate if present, reacts with potassium iodide in the presence of acid and liberates iodine which will give the blue colour.



PROCEDURE:

Weigh 0.5 g of the sample accurately and dissolve in 10 ml of CO₂ Free water in a boiling tube. Add 0.15 ml of dilute sulphuric acid from a 1 ml graduated pipette and one drop of iodine free starch solution.

OBSERVATION

No blue colour is produced within 2 minutes.

REPORT

The sample passes the test.

Ex No: 15

Date:

SWELLING POWER IN BENTONITE

AIM:

To perform the test for purity in swelling power on bentonite.

PRINCIPLE:

When water is added to bentonite, each particle is suspended by a layer (or) shell of water. This produces a particle several times larger than the original particle. Swelling of mass results. Bentonite can adsorb up to 5 times its weight of water and its bulk may increase by twelve to fifteen times. Bentonite is insoluble in water but swells into a homogeneous mass.

To find out the swelling power, the sample is added in small quantities at intervals of 2 mins to a solution of sodium lauryl sulphate in a 100 ml measuring cylinder. It is allowed to stand for 2 hours. The presence of wetting agent (sodium lauryl sulphate) promotes compatibility between the insoluble bentonite and water.

PROCEDURE:

Dissolve 1 gm of sodium lauryl sulphate in 100 ml of water and transfer to a 100 ml graduated or measuring cylinder having a diameter of 3 cm.

Weigh accurately 2 gm of the sample of bentonite and add it in small quantities every 2 minutes to the above solution. Allow each portion to settle. Set aside for 2 hours find out the apparent volume of the sediment at bottom of the cylinder.

OBSERVATION

The apparent volume of sediment is 24 ml.

REPORT

The sample passes the test.

Ex No: 16

Date:

IDENTIFICATION TEST FOR FERROUS SULPHATE

AIM:

To identify the given sample and report it.

PROCEDURE:

S.NO.	EXPERIMENT	OBSERVATION	INFERENCE
1.	Test for Ferrous: I) Transfer 10 mg of the substance into a test tube and add 1ml of Potassium ferrocyanide solution. ii) Transfer 10 mg of the substance into a test tube and add 1ml of Potassium ferric cyanide solution.	White precipitate rapidly becomes blue and insoluble in dilute hydrochloric acid. Dark blue precipitate. Insoluble in dilute hydrochloric acid. Decompose in sodium hydroxide.	Presence of ferrous. Presence of ferrous.
2.	Test for Sulphate: i) Transfer 50mg of the substance into a test tube and add 5ml water. Add 1ml of hydrochloric acid and 1ml of barium chloride solution. ii) Transfer 50mg of the substance into a test tube and add 5ml water. Add 2ml of lead acetate solution.	White precipitate is formed White precipitate soluble in ammonium acetate and in sodium hydroxide solution.	Presence of Sulphate. Presence of Sulphate.

REPORT:

The given sample contains i) Ferrous (cation)

ii) Sulphate (anion)

Ex No: 17

Date:

IDENTIFICATION TEST FOR COPPER SULPHATE

AIM:

To identify the given sample and report it.

PROCEDURE:

S.NO.	EXPERIMENT	OBSERVATION	INFERENCE
1.	Test for Copper: I) To the test solution taken in a test tube and add sodium hydroxide solution. ii) Transfer the test substance into a test tube and add dil.acetic acid and potassium iodide solution.	Light blue precipitate is formed. Boil the precipitate it becomes brownish black. Brownish precipitate is formed. This gives a deep blue colour with starch solution.	Presence of Copper. Presence of Copper.
2.	Test for Sulphate: i) Transfer 50mg of the substance into a test tube and add 5ml water. Add 1ml of hydrochloric acid and 1ml of barium chloride solution. ii) Transfer 50mg of the substance into a test tube and add 5ml water. Add 2ml of lead acetate solution.	White precipitate is formed White precipitate soluble in ammonium acetate and in sodium hydroxide solution.	Presence of Sulphate. Presence of Sulphate.

REPORT:

The given sample contains i) Copper (cation)

ii) Sulphate (anion)

Ex No: 18

Date:

IDENTIFICATION TEST FOR CALCIUM GLUCONATE

AIM:

To identify the given sample and report it.

PROCEDURE:

S.NO.	EXPERIMENT	OBSERVATION	INFERENCE
1.	<p>Test for Calcium:</p> <p>I) Transfer 20 mg of the test substance into a test tube and add 5ml of acetic acid and 0.5ml potassium ferro cyanide solution.</p> <p>ii) Transfer 20 mg of the substance into a test tube and add 1ml water and 0.2ml of 2% w/v solution of ammonium oxalate.</p>	<p>White crystalline precipitate is obtained.</p> <p>White precipitate. Sparingly soluble in dil. Acetic acid Soluble in dilute hydrochloric acid.</p>	<p>Presence of Calcium.</p> <p>Presence of Calcium.</p>
2.	<p>Test for Gluconic Acid:</p> <p>i) Transfer 1ml of the test substance into a test tube and add few drops of ferric chloride solution.</p> <p>ii) Transfer 0.25gm of the test substance into a test tube and add 2.5ml of warm water. Add 1ml glacial acetic acid and freshly distilled 1.5ml phenylhydrazine. Heat the mixture in a water bath for half an hour and allow to cool. Scratch the inner surface of the test tube with a glass rod until crystals of gluconic acid phenylhydrazine begin to form. Dissolve the precipitate in 10ml hot water, add small amount</p>	<p>Yellow colour appears.</p> <p>A white colour crystal is obtained.</p>	<p>Presence of Gluconic Acid.</p> <p>Presence of Gluconic Acid.</p>

	of decolourising charcoal and filter.		
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REPORT:

The given sample contains i) Calcium (cation)

ii) Gluconate (anion)

Date:

IDENTIFICATION TEST FOR AMMONIUM CHLORIDE

AIM:

To identify the given sample and report it.

PROCEDURE:

S.NO.	EXPERIMENT	OBSERVATION	INFERENCE
1.	Test for Ammonium: I) Transfer 10 mg of the test substance into a test tube and add 1ml of sodium hydroxide solution. Heat. ii) Transfer 100 mg of the test substance into a test tube and add 1ml water. Add 0.2mg of light magnesium oxide. Add 1ml of hydrochloric acid 0.05ml of methyl red solution.	Ammonia vapours, turns red litmus to blue. Colour of solution change to yellow.	Presence of Ammonium. Presence of Ammonium.
2.	Test for Chloride: i) Transfer small quantity of the substance into a test tube and add 2ml water. Acidify with dil. nitric acid. Add 0.5ml of silver nitrate solution. Shake and allow to stand. ii) Transfer small quantity of the substance into a test tube and add 0.2g of potassium dichromate and 1ml sulphuric acid. Place a filter paper strip moistened with 0.1ml of diphenyl carbazide solution over the mouth of the test tube.	Curdy white precipitate is formed. Insoluble in dil. Nitric acid but soluble in dil. ammonia Paper turns violet to red.	Presence of Chloride. Presence of Chloride.

REPORT:

The given sample contains i) Ammonium (cation)

ii) Chloride (anion)