Vidya Siri College of Pharmacy

#67/4, Off Sarjapur Road, Bangalore East Taluk, Chikkakannalli, Bengaluru – 560 035

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VIDYA SIRI
COLLEGE OF PHARMACY

PHARMACEUTICAL CHEMISTRY
LABORATORY RECORD

Name of the Student :
.....

Reg. No. : .....

Class :.....

Batch : .....

Vidya Siri College of Pharmacy
#67/4, Off Sarjapur Road, Bangalore East Taluk, Chikkakannalli, Bengaluru – 560 035

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VIDYA SIRI
COLLEGE OF PHARMACY

CERTIFICATE

This is to certify that Mr./Ms.

_____

is a student of ***D.Pharm PART-I (ER 2020)****and has satisfactorily completed the Practical prescribed by Board of Examination Authority, Bangalore in* ***PHARMACEUTICALCHEMISTRY*** *during the academic year.....Reg. No.____Date:___*

Signature of the Subject Teacher

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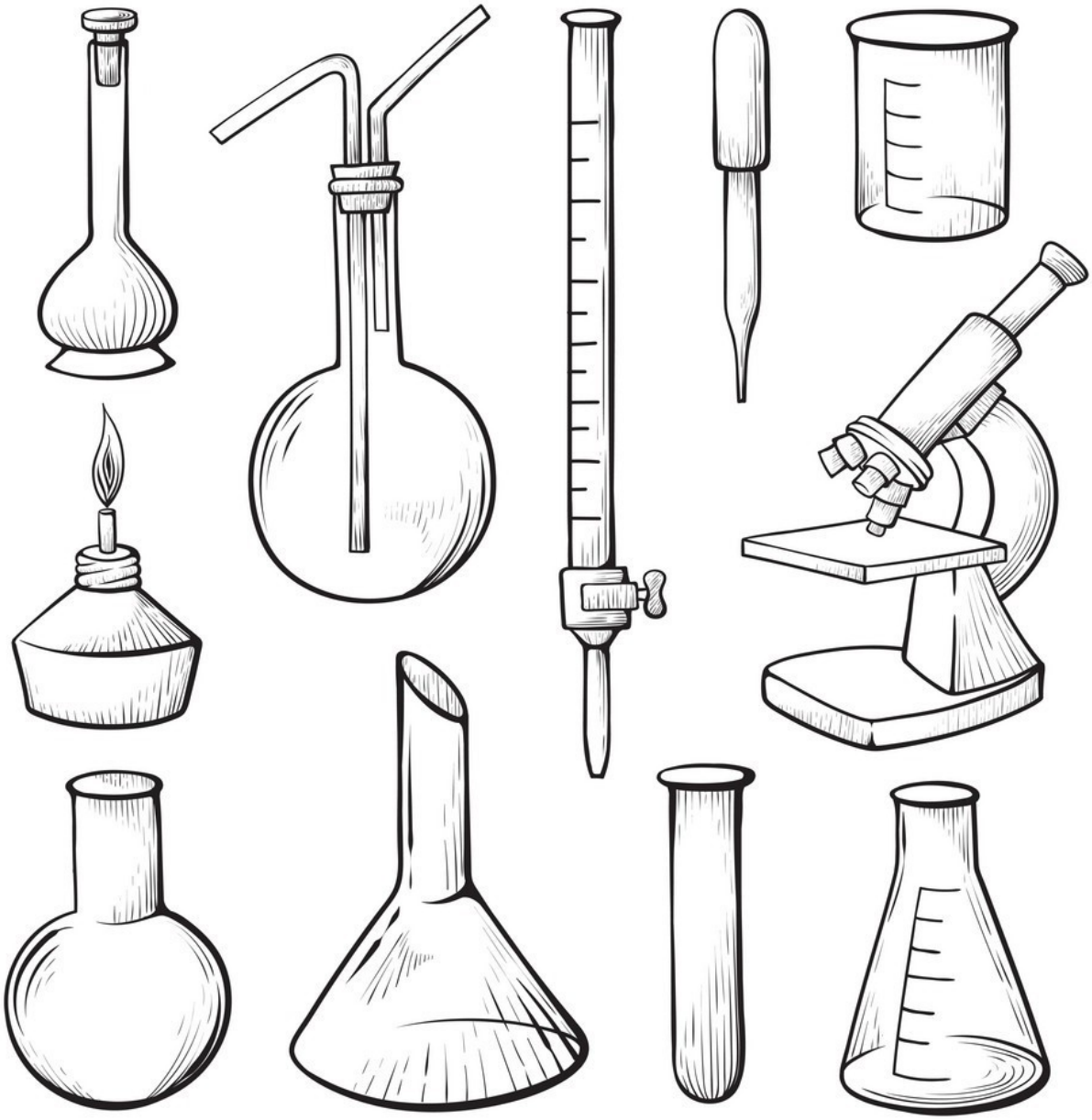
26		To analyze the presence of acid radicals (anions) in the given mixture.	

GLASS –WARE USED IN CHEMISTRY LAB



EXPERIMENT NO. 1 **DATE:**

TO STUDY THE GENERAL GLASSWARE USED IN LAB

Aim: To study the general glassware used in lab.

Requirements: Apparatus Required: Beaker, measuring cylinder, conical flask, burettes, burette stand, volumetric flask, spatula, watch glass, etc

Theory: General glassware's and their uses:

1. Burette: A piece of glassware used for carefully measured volume of a liquid reagent comes in various sizes, consists of calibrated tube. These are available in capacities of 1-100 ml. These are long graduated tubes of uniform bare and are closed at lower end by a stopcock. These are also used to store corrosive reagents.

2. Pipettes: A piece of glassware used for measuring the volume of liquid comes in various sizes. All pipettes are designed for transfer of known volumes of liquids from one container to another. We have two types of pipettes:

(a) Volumetric or transfer pipette: Have one calibration mark and are used to deliver a single specific volume or fixed volumes of liquids, quite accurately.

(b) A measuring or graduated pipette or Mohr pipette: It is graduated along with its length in convenient units.

3. Graduated cylinder: A piece of glassware used to measure approximate volume of liquids.

4. Volumetric flask: A piece of glassware manufactured with capacities ranging from 5 ml to 5 litres and is usually calibrated to contain the specified volume when filled to the calibration ring on the neck.

5. Conical flask (Erlenmeyer flask): It is a piece of glassware comes in various sizes used for: a) titration b) Filtration c) Distillation d) Heating and evaporation.

6. Beaker: A piece of glassware comes in various sizes used for.

(a) Dissolving the samples.

(b) Transfer the solution.

7. Stirring glass rod: A piece of glassware in various sizes used for:

(a) In solubility mixed solute with solvent.

(b) In decantation.

(c) Collecting the ppts.

8. Watch glass: A piece of glassware in various sizes using in:

(a) Weighing non-hygroscopic material

b) Covering the beaker that contains heat solution.

(c) Covering the beakers during heat solution.

9. Spatula: A piece of lab tool used for transfer of solid material from a reagent bottle, manufactured from steel and ceramic materials. 10. Brush: A piece of lab tool used for cleaning the glassware in various sizes.

11. Burette stand: It is also called as clamp stand intended to support various equipment and glasswares like; burettes, test tubes and flasks.

12. Clamp: It is scientific equipment used to hold and secure a burette on a stand.

13. Funnel:

A piece of glassware used for.

(a) Transfer the liquid

(b) Filtration

- **Result:** Hence, the general glassware's used in the lab were well studied.

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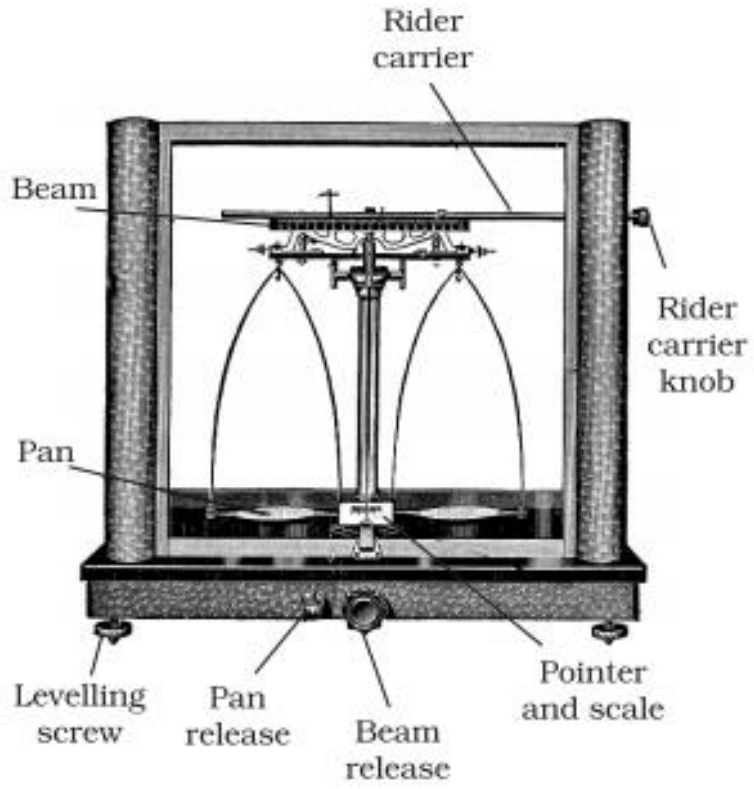
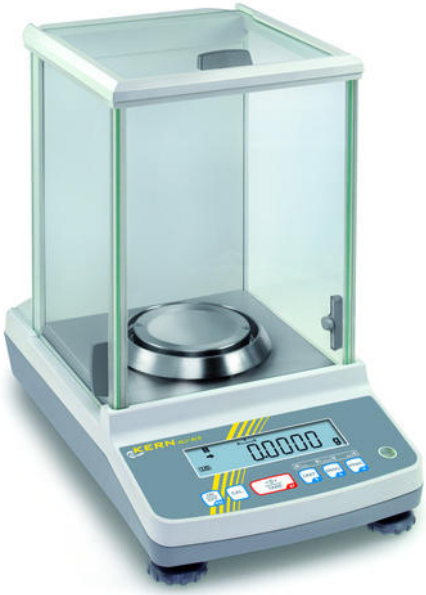


Analytical balance



Digital balance

EXPERIMENT NO. 2 **DATE:**

TO STUDY THE BALANCES USED IN LABORATORY

Aim: To study the balances used in laboratory. Requirements:

Apparatus: Weight box, balance, watch-glass, material to be weighed, etc.

Theory: The laboratory instrument built for measuring weight is called the balance. The name is derived from mechanical devices that utilize known weights to balance the object to be weighed across a fulcrum. Quantitative analysis involves weighing with great accuracy. Analytical balance in quantitative analysis can be used for weighing substances not heavier than 200 gm. The least count is 0.0002 gm. An electronic balance is one that uses an electromagnet to balance the object to be weighed on a single pan. A chemical can be conveniently weighed on a piece of weighing paper without having to determine the weight of the paper. Tarring means that the balance is simply zeroed with the weighing paper on the pan.

The balance is enclosed in a glass case which protects it from dust, air movements, etc. The base of the balance rests on screws whereby the edges and agate plates on which they rest are brought into horizontal position by means of plumb bob attached to the balance column. The balance pans are made of light metal coated with nickel to prevent oxidation. Substances should not be placed on pans directly. It should be weighed on watch glasses or in weighing bottles.

A balance that is used to obtain four or five digits to the right of the decimal point in the analytical laboratory is called the analytical balance. The modern laboratory utilizes single pan electronic analytical balance almost exclusively for precision. In the weight box, weights are arranged in the order of 50, 20, 20, 10, 5, 2, 2, 1 and it also has a rider made of thin wire. The weight of rider is 0.0002 gm. The rider is placed on the beam with the help of a hook which is fitted with the balance.

Procedure:

1. Raise the beam slowly and note that the pointer swings equal decisions on both sides of the zero mark.

2. Adjust the balance before taking weights on the pan.

3. Always take the weight of permitted load and never overload the balance.

4. If the weighing substances are wet use watch glass or weighing bottle. Do not place wet substance directly on the pan.

5. Do not weigh if the substance is hot.

6. While weighing only use side doors of the balance**.**

7. The weights, fractional weights or rider should not be touched directly with finger.

8. Always use the same balance for weighing different substances to reduce error.

• **Rider:** The riders are the sliding pointers positioned on top of the beams to show the pan and beam weight in grams. It is a loop of a small aluminum wire and usually weighs 10 mg. It can be easily seated on the balance beam.

Result: Hence, the study of balances has been carried out successfully.

Observations for Chloride:

Standard Solution	Test Solution
More opalescence	More opalescence
Equal opalescence	Equal opalescence
Less opalescence than standard solution	Less opalescence than test solution

Inference:

The opalescence of both solutions is compared. The given compound a passed/o did not pass the limit test for chloride.

Passed-Due to less opalescence

Did not pass-Due to more opalescence

EXPERIMENT NO. 3 **DATE:**

TO PERFORM LIMIT TEST FOR CHLORIDE IN GIVEN COMPOUND

Aim: To perform limit test for chloride in given compound.

Requirements:

Chemicals: Chloride standard solution (25 ppm), dilute nitric acid, distilled water, 0.1 M silver nitrate solution. **Apparatus:** Nasser's cylinder, glass rod, beaker, dropper, volumetric flasks (100 ml), pipette (10 ml), etc.

Theory: Limit tests are performed to check the limits of impurities present in pharmaceutical substances. These tests involve comparison of opalescence, turbidity or colour of solutions of test substances with standard substances. The extent of opalescence, turbidity, and colour is affected by presence of impurities present in substance, variation in time and method of performance of test. Limit test for chloride is based on reaction of silver nitrate with soluble chlorides to form precipitate of silver chloride which is insoluble in dilute nitric acid. The extent of precipitate depends upon the amount of silver chloride formed or amount of chloride ions present in the substance. The opalescence produced is compared with standard opalescence from standard solution containing a fixed amount of chloride under same experimental conditions. The opalescence produced by test substance should be less intense than that of standard substance to pass the limit test for chloride.

$$NaCl + AgNO_3 \xrightarrow{\text{Dil. } HNO_3} AgCl + NaNO_3$$

White ppt.

Procedure:

1. Preparation of chloride standard solution: Dilute 5 volumes of 0.0824% w/v of sodium chloride solution to 100 volumes with water. It gives standard chloride solution of 25 ppm.

2. Preparation of test opalescence: Dissolve the specified quantity of the substance under examination in water, or prepare a solution as directed in the individual monograph and transfer it to a Nessler's cylinder. Add 10 ml of dilute nitric acid, except when nitric acid is used in the preparation of the solution, dilute to 50 ml with water and add 1 ml of 0.1 M silver nitrate, Stir immediately with a glass rod and allow to stand for 5 minutes protected from light.

3. Preparation of standard opalescence: Take a mixture of 10.0 ml of chloride standard solution (25 ppm Cl) and 5 ml of water in a Nessler's cylinder. Add 10 ml of dilute nitric acid,

dilute it to 50 ml with water and add 1 ml of 0.1 M silver nitrate. Stir immediately with a glass rod and allow to stand for 5 minutes protected from light.

4. Observation: After 5 minutes, view both standard and test cylinder transversely against a black background.

Result:

The opalescence of both solutions is compared. The given compound □ passed / □ did not pass the limit test for chloride.

Standard Solution	Test Solution
More opalescence	More opalescence
Equal opalescence	Equal opalescence
Less opalescence than standard solution	Less opalescence than test solution

Observations for Sulphate:

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Inference:

The opalescence of both solutions is compared. The given compound a passed/o did not pass the limit test for sulphate.

- Passed-Due to less opalescence
- Did not pass-Due to more opalescence

EXPERIMENT NO. 4 **DATE:**

TO PERFORM LIMIT TEST FOR SULPHATE IN GIVEN COMPOUND

Aim: To perform limit test for sulphate in given compound.

Requirements:

Chemicals: Standard sulphate solution (10 ppm), ethanol standard sulphate solution (10 ppm), 25% w/v barium chloride solution, 5 M acetic acid, distilled water. Apparatus: Nessler's cylinder, glass rod, beaker, dropper, volumetric flasks (100 ml), pipette (1 and 10 ml), etc.

Theory: Limit test for sulphate is based on reaction of barium chloride and soluble sulphate in acidic solution. The opalescence formed in test solution is compared with the standard opalescence obtained by the fixed quantity of the sulphate under the same experimental conditions. Test solution may contain small amount of potassium sulphate, which increases the sensitivity of test. Any sulphate ion impurity in test substance will produce barium sulphate in excess of already dissolved amount, causing turbidity. To prevent super saturation of barium sulphate, which may occur, small amount of ethanol is added. The opalescence produced by test substance should be less intense than that of standard substance to pass the limit test for chloride.

$$BaCl_2 + Na_2SO, \Longrightarrow BaSO4+ + 2NaCI$$

White ppt.

Procedure:

1. Preparation of ethanolic sulphate standard solution: Dilute 1 volume of 0.181% w/v of potassium sulphate solution in 30% ethanol to 100 volumes with 30% ethanol. It gives ethanolic standard sulphate solution of 10 ppm.

2. Preparation of sulphate standard solution: Dilute 1 volume of 0.181% w/v ofpotassium sulphate solution in distilled water to 100 volumes with distilled waterIt gives standard sulphate solution of 10 ppm.

3. Preparation of test opalescence: To 1.0 ml of a 25.0 % w/v solution of barium chloride in a Nessler's cylinder, add 1.5 ml of ethanolic sulphate standard solution (10 ppm SO4), 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 solution. Stir immediately with a glass rod and allow to stand for 5 minutes.

4. Preparation of standard opalescence: To 1.0 ml of a 25.0 % w/v solution of barium chloride in a Nessler's cylinder, add 1.5 ml of ethanolic sulphate standard solution (10 ppm SO). Mix and allow to stand for 1 minute. Add 15 ml of Pharmaceutical Chemistry (F.Y. D. Pharm) sulphate standard solution (10 ppm SO) in 15 ml of water and 0.15 ml of 5 M acetic acid. Add sufficient water to produce 50 ml solution. Stir immediately with a glass rod and allow to stand for 5 minutes.

5. Observation:

After 5 minutes, view both standard and test cylinder transversely against a black background.

Result:

The opalescence of both solutions is compared. The given compound o passed/a did not pass the limit test for sulphate.

More opalescence	More opalescence
Equal opalescence	Equal opalescence
Less opalescence than standard solution	Less opalescence than test solution

Observations for Iron:

Inference:

The opalescence of both solutions is compared. The given compound a passed/o did not pass the limit test for iron.

- Passed-Due to less opalescence
- Did not pass-Due to more opalescence

EXPERIMENT NO. 5 **DATE:**

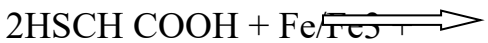
TO PERFORM LIMIT TEST FOR IRON IN GIVEN COMPOUND

Aim: To perform limit test for iron in given compound**.**

Requirements:

Chemicals: Iron standard solution (20 ppm), 20% w/v iron free citric acid, thioglycolic acid iron free ammonia solution, distilled water

Apparatus: Nessler's cylinder, glass rod, beaker, dropper, volumetric flasks (100 ml).pipette (10 ml), etc. **Theory:** Limit tests are performed to check the limits of impurities present in pharmaceutical substances. These tests involve comparison of opalescence, turbidity or colour of solutions of test substances with standard substances. The extent of opalescence, turbidity, and colour is affected by presence of impurities present in substance, variation in time and method of performance of test. Limit test for iron is based on reaction of iron in ammoniac solution in the presence of citric acid with thioglycolic acid to form ferrous thioglycolate. The ammoniac ferrousthioglycolate solution is pink to deep reddish purple color depending on concentration of iron. The intensity of colour is compared with standard intensity obtained from fixed concentration of iron. Iron may be precipitated as ferrous or ferric hydroxide on reaction with ammonia solution. The precipitate may interfere with the intensity of colour. Citric acid prevents the precipitation of iron as hydroxide by forming complex with iron which is not precipitated by ammonia solution



2HSCH COOH + Fe/Fe3 +

Fe(HSCH2COOH)+2H+

Thioglycolic acid Ferrous thioglycolate

Procedure:

1. Preparation of iron standard solution: Dilute 1 volume of 0.1726% w/v of ferric ammonium sulphate in 0.05 M sulphuric acid to 10 volumes with water. It give standard iron solution of 20 ppm.

2. Preparation of test colour: Dissolve the specified quantity of the substance under examination in 40 ml water, or prepare a solution (10 ml) as directed in the monograph, and

transfer to a Nessler's cylinder. Add 2 ml of a 20% w/v solution of iron-free citric acid and 0.1 ml of thioglycolic acid. Mix them and make alkaline with iron-free ammonia solution. Dilute it to 50 ml with water and allow standing for 5 minutes.

3. **Preparation of standard opalescence:** Take 2 ml of iron standard solution (20 ppm) in a Nessler's cylinder. Add 2 ml of a 20% w/v solution of iron-free citric acid and 0.1 ml of thioglycolic acid. Mix them and make alkaline with iron-free ammonia solution. Dilute it to 50 ml with water and allow to stand for 5 minutes.

4. Observation: After 5 minutes, view both standard and test cylinder to compare intensity of colour produced.

Result:

The opalescence of both solutions is compared. The given compound passed/o did not pass the limit test for iron.

More opalescence	More opalescence
Equal opalescence	Equal opalescence
Less opalescence than standard solution	Less opalescence than test solution

Observations for Heavy metals:

Inference:

The opalescence of both solutions is compared. The given compound a passed/o did not pass the limit test for heavy metals.

- Passed-Due to less opalescence
- Did not pass-Due to more opalescence

EXPERIMENT NO. 6 **DATE:**

TO PERFORM LIMIT TEST FOR HEAVY METALS IN GIVEN COMPOUND

Aim: To perform limit test for heavy metals in given compound.

Requirements:

Chemicals: Lead nitrate, dilute acetic acid, dilute ammonia solution, hydrogen sulphide solution. Apparatus: Nessler's cylinder, glass rod, beaker, dropper, volumetric flasks, pipette.

Theory: Limit test for heavy metals is designed to determine the content of metallic impurities that are coloured by sulphide ion, under the specified conditions. The limit for heavy metals is indicated in the individual monograph in terms of part of lead (Pb) per million parts (by weight) of substance with that of control prepared from standard lead solution. The amount of heavy metal is determined by one of the following methods directed in the individual monograph.

Procedure:

1. Preparation of lead standard solution:

(**a) Lead standard solution (0.1% Pb):** Dissolve 0.400 gm of lead nitrate in water containing 2 ml of nitric acid and add sufficient water to produce 250 ml solution.

(**b) Lead standard solution (100 ppm Pb):** Dilute 1 volume of lead standard solution (0.1% Pb) to 10 volumes with water.

(c) Lead standard solution (20 ppm Pb): Dilute 1 volume of lead standard solution(100 ppm Pb) to 5 volumes with water.

Method A:

Standard solution: Into a 50 ml Nessler's cylinder pipette 1.0 ml of lead standard solution (20 ppm Pb) and dilute it with water to 25 ml. Adjust with dilute acetic acid or dilute ammonia solution to a pH between 3.0 and 4.0. Dilute with water to about 35 ml and mix.

Test solution: Into a 50 ml Nessler's cylinder, place 25 ml of the solution prepared for the test as directed in the individual monograph or dissolve the specified quantity of the substance under examination in sufficient water to produce 25 ml solution. Adjust with dilute acetic acid

or dilute ammonia solution to a pH between 3.0 and 4.0, dilute it with water to about 35 ml and mix.

Procedure: To each of the cylinders, containing the standard solution and test solution respectively, add 10 ml of freshly prepared hydrogen sulphide solution, mixit, and dilute to 50 ml with water. Allow to stand for 5 minutes and view downwards over a white surface.

Method B:

Procedure: Transfer 12 ml of each of the test solutions prepared as described under preparations of the test solutions to a beaker, add 2 ml of acetate buffer, pH 3.5, TS and mix. Add 1.2 ml of freshly prepared thioacetamide reagent TS, mix and allow to stand for two minutes. Filter the solutions through a suitable membrane filter (nominal pore size 0.45 µm). Carry out the filtration slowly and uniformly, applying moderate and constant pressure to the piston. Compare the intensity of the coloration of the residues obtained with the different test solutions on the membrane filters. The test is not valid unless.

Result:

The opalescence of both solutions is compared. The given compound o passed/o did not pass the limit test for lead (heavy metal).

Experiment	Observations	Inference
Colour:	Blue or bluish green Greenish Light green Dark brown Pink Light Pink White colourless	Cu2+ OR Ni 2+ Ni2+ Fe2+ Co2+ Mn2+ Shows absences of cu2+,Ni 2+, Fe2+,Co2+,Mn2+.
Smell: Take a pinch of the salt between your Vinegar like smell Ammonical smell fingers and rub with a drop of water.	Ammonical smell Vinegar smell Smell like that of rotten eggs Heavy Light fluffy powder	NH4+ CH3COO- S2-
Density	Heavy light fluffy powder	Salt of pb2+ or Ba2+ carbonate

Apperance - Nature

Crystalline	Water soluble salts Le salts of group VI radicals, salts containing Cl, B r- , SO{4} 2- , N O2- , N O 3-	
Amorphous	Water insoluble salts ie ,Co 3- ,s2 , PO 3- etc.	
Hygroscopic	Nitrates, Nitrites,Chlorides, of C_{v} Zn, Mn, Mg, Ca, etc.	

EXPERIMENT NO. 7 **DATE:**

IDENTIFICATION TESTS FOR ANIONS AND CATIONS AS PER INDIAN PHARMACOPOEIA

Aim: Identification tests for anions and cations as per Indian Pharmacopoeia

Requirements:

Apparatus: Test tubes, Boiling Tubes Test tube holder, Test tube stand, Corks, Filter Paper, Delivery Tube. **Chemicals:** Barium sulphate, Calcium phosphate, sodium carbonate, silver chloride,

Identification tests for Cations:

1. Silver ion (Ag): Silver is a metal element, which exists in compounds asmonovalent cation. Although many silver salts are sparingly soluble, silver nitrate is easily soluble in water. Silver nitrate is used in medicine in dermatologists practice as a lunar caustic

Detection of Ag': Hydrochloric acid or soluble chlorides precipitate white silver chloride sediment from solutions containing soluble silver salts (e .g. silver nitrate). Silver chloride can be solubilized in ammonium hydroxide yielding complex compound diamine silver chloride.

Procedure: Add 0.5 * 1m / o silver salt solution to a test tube and then add 2 drops of 2M HCI. Observe formation of AgCl white precipitate. Take a portion of the precipitate and transfer to another test tube. Add the N*H_{3} .H 2 O solution in excess. The precipitate will dissolve. AgCl precipitate can be obtained again on addition of nitric acid to the solution

$$[Ag(NH),]CI + 2\ HNO,\ AgCl + 2\ NH,NO_1$$

2. Calcium ion (C a 2+); Calcium cation, present like barium in the second group of elements in the periodic table, forms bivalent ions. Calcium compounds are commonly found in nature as minerals, mostly in forms of calcium carbonate CaCO3 (chalk), hydrated calcium sulphate (gypsum) and semi-hydrate calcium sulphate. Calcium phosphate forms a main stiff structural compound of bone. Calcium ions. play important roles in metabolic processes. In human organism calcium comprises 1.5 \% of the total body weight.

Detection of C a2+ ; While heating the platinum wire with calcium salt solution in aflame, as the result of calcium ion electrons excitation, change the flame color to reddish-brown is observed.

Procedure: Anneal the platinum wire in the laboratoryit back burner flame until the flame becomes Colourless Then immerse the wire in calcium salt solution and to the flame again Observe the change of Colour flame to reddish-brown. put sparingly X-ray agent

3. Barium ion (Ba): Barium is a heavy metal which forms bivalent compounds. A water-soluble barium salts are toxic Barium sulphate is not toxic, it is soluble in water, acids and bases Barium sulphate strongly absorbs the radiation and is used in medical examination as a contrasting

Detection of Ba2+: Suphuric acid and its soluble salts form abundant white sediment roentgenoscopy of barium sulphate after mixing with barium cations containing solutions.

$$BaCl, H,SO \Longrightarrow BaSO\text{-}2HC1$$

Procedure 1: Add 2 drops of IM sulfuric acid (or gypsum water) to 0.5-1.0 ml of BaSO, + Cack barium sulfate with solutions of strong acid (HCI) and strong base (NaOH). While heating the platinum wire with barium salt solution in a flame, as the result of barium ion electrons excitation, change the flame Colour to green is observed.

Procedure 2: Anneal the platinum wire in the laboratory burner flame until the flame becomes Colourless. Then, dip the wire into the barium salt solution and put it backinto the flame. Observe a green Colour of the flame.

4. Sodium ion (Na'): Sodium is a human body extracellular element. It is commonly present in living organisms and in various minerals as monovalent ion Almost all sodium salts are easily soluble in water. Sodium ion can be easily excitated in the temperature of laboratory burner flame, resulting in visible strong yellow light emission. It does not form characteristic insoluble salt sediments, so it can beidentified and quantitatively measured using a flame test.

Detection of Na':

Procedure: Anneal the platinum wire in the laboratory burner flame until the flame becomes colourless. Next dip the wire into the sodium salt solution and put it back to the flame. Observe the change of the flame Colour to strong yellow.

5. Potassium ion (K'): Potassium is human body intracellular element. It is commonly present in living organisms and in various minerals as monovalent ion

Detection of K': Procedure: Anneal the platinum wire in the laboratory burner flame until the flame becomes colourless. Next dip the wire into the potassium salt solution and put it back to the flame. Observe the change of the flame Colour to violet.

Silver nitrate (AgNO,) reacts with chloride solution to precipitate white silver chloride, Silver chloride is soluble in excess of NH, HO, in solutions of Na,S,O KCN and concentrated HCI,

Detection of CI:

NaCl + AgNO, AgCl + 2NH, H,O

AgCl + NaNO, [Ag(NH)2]CI+ $2H_2O$

• **Procedure**: Add a few drops of silver nitrate solution to a test tube containing 0.5. Di-ammine silver chloride 10 ml of chloride anions solution. As a result AgCI precipitate is formed. Transfer a portion of the precipitate to another test tube and add drop by drop 2 M NH3 H2O solution, until the precipitate is dissolved. AgCl can be re-precipitated again upon addition of nitric acid.

• **Result:**

Observation

Standardization Of 1 M Hydroxide Solution

Sr.no	Volume of oxalic acid taken (ml)	Burette reading (ml) intial	Burette reading (ml) final	Volume of NaoH used (Ml)
1	**10**			
2	**10**			
3	**10**			

Average volume of NaOH used (V)= ml

Average volume of oxalic acid taken (v2)=10ml

Calculations:

The morality of given 1 M sodium hydroxide solution is calculated by using the following formula:

M1V1/n1=M2V2/n2

M1= Morality of NaOH

M2= Morality of oxalic acid =1M

V2= Volume of oxalic acid = 10 ml

V1= Volume of NaOH

n1=1 n2=1

M1= n1M2V2/n2V= 1x1x10/1 x V1 =10/…… =……M

Inference: The exact morality of given sodium hydroxide solution is M(P) NaOH……..M

EXPERIMENT NO. 8 **DATE:**

TO PREPARE AND STANDARDIZE SODIUM HYDROXIDE SOLUTION.

Aim: To prepare and standardize sodium hydroxide solution.

Requirements:

Chemicals: 0.1 M sodium hydroxide solution, 0.1 M oxalic acid solution. phenolphthalein indicator, distilled water. Apparatus: Burette, pipette (10 ml), conical flask, beaker, volumetric flask (1000 ml), funnel, glass rod.

Theory: Sodium hydroxide is a strong base which has molecular weight 40 gm/mol. It is hygroscopic in nature and hence cannot be weighed properly. Oxalic acid is a weak acid and has molecular weight 126 gm/mol and is a primary standard. Sodium hydroxide can be standardized by titrating with oxalic acid solution. This titration is an example of acidimetry in which base is titrated with an acid. The following reaction takes place in this titration:

$$H_2C_2O+2NaOH \rightarrow 2COONa+ 2H_2O$$

Procedure:

1. Preparation of 0.1 M sodium hydroxide solution: Weigh accurately 4 gm of sodium hydroxide pellets and put into a 250 ml beaker. Add approximately 150 ml of distilled water in it and stir it to dissolve the pellets. Pour this solution to a volumetric flask (1000 ml) and make up the volume. Shake well to mix properly.

2. Preparation of 0.1 M oxalic acid solution: Weigh accurately 12.6 gm of oxalic acid and put it into a beaker (250 ml). Add approximately 150 ml of distilled water and stir it to make clear solution. Transfer this solution to a volumetric flask (1000 ml) and make up the volume. Shake well to mix properly.

3. Titration: Rinse and fill the burette with 0.1 M sodium hydroxide solution with the help of funnel. Pipette out 10 ml of 0.1 M oxalic acid solution into a clean and dry conical flask and add 2-3 drops of phenolphthalein indicator to it. Shake it to mix the contents. Titrate the contents of conical flask with 0.1 M sodium hydroxide solution until permanent pink colour is obtained. Repeat the titration and take three concordant readings. Calculate molarity of 0.1 M sodium hydroxide solution.

• **Result:** The exact molarity of given sodium hydroxide solution is ___M

Observation

Standardization Of 1 M potassium permanganate Solution

Sr.no	Volume of Oxalic acid taken (ml)	Burette reading (ml) intial	Burette reading (ml) final	Volume of potassium permanganate used (Ml)
1	**10**			
2	**10**			
3	**10**			

Average volume of potassium permanganate (V1)= ml

Average volume of Oxalic Acid (v2)=10ml

Calculations:

The morality of given 1 M potassium permanganate solution is calculated by using the following formula:

M1V1/n1=M2V2/n2

M1= Morality of potassium permanganate

M2= Morality of oxalic acid =0.1M

V2= Volume of oxalic acid = 10 ml

V1= Volume of potassium permanganate

n1=2 n2=5

M1= n1M2V2/n2V= 1x1x10/1 x V1 =10/…… =……M

Inference: The exact morality of given potassium permanganate solution is M(P) KMnO4……..M

EXPERIMENT NO. 9 **DATE:**

TO PREPARE AND STANDARDIZE POTASSIUM PERMANGANATE SOLUTION

Aim: To prepare and standardize potassium permanganate solution,

Requirements:

Chemicals: 01 M potassium permanganate, 0.1 M oxalic acid solution, dilute sulphuric acid, distilled water**.**

Apparatus: Burette, pipette (10 ml), conical flask, beaker, volumetric flask (1000 ml). funnel, glass rod.

Theory: Potassium permanganate is a strong oxidizing agent which has molecular weight 158 gm/mol. Potassium permanganate contains not less than 99.0% and not more than 100.5% of KMnO4, Oxalic acid is a weak acid and has molecular weight 126 gm/mol and is a primary standard. Potassium permanganate can be standardized by titrating with oxalic acid solution. This titration is an example of redox titration. The temperature is maintained at 60-70°C during this reaction as reaction proceeds slowly at room temperature. No indicator is required in this titration as KMnO4 is self-indicator. Colour change takes place from colourless to faint pink. The following reaction takes place in this titration

$$2KMnO3H,SO_1 + 5H_2C_2O_1 \qquad K_2SO4 + 2MnSO4 + 8H_2O + 10CO2$$

Procedure:

1. Preparation of 0.1 M potassium permanganate solution: To prepare 0.1 M solution, dissolve 15.8 gm of potassium permanganate in 900 ml of water and heat it on a water-bath for 1 hour. Then cool and filter through a sintered-glass filter and add sufficient water to produce 1000 ml solution. Store and protect from moisture and sun light.

2. Preparation of 0.1 M oxalic acid solution: Weigh accurately 12.6 gm of oxalic acid and put it into a beaker (250 ml). Add approximately 150 ml of distilled water and stir it to make clear solution. Transfer this solution to a volumetric flask (1000 ml) and make up the volume. Shake well to mix properly.

3**. Titration:** Rinse and fill the burette with 0.1 M potassium permanganate solution with the help of funnel. Pipette out 10 ml of 0.1 M oxalic acid solution into a clean and dry conical flask

and add 10 ml of dilute sulphuric acid. Shake it well to mix the contents. Heat the contents of conical flask to 60-70°C. Titrate the contents of conical flask with 0.1 M potassium permanganate solution until permanent faint pink colour is obtained. Repeat the titration and take three concordant readings. Calculate morality of 0.1 M potassium permanganate solution.

Result: The exact morality of given potassium permanganate solution is……..M

• **Assay of Ferrous sulphate:**

Sr.no	Volume of FeSO solution (ml)	Burette reading (ml) Initial	Burette reading (ml) Final	Volume of KMnO, used (ml)
1	20			
2	20			
3	20			

Average volume of KMnO, used =……..ml

• **IP factor or equivalent factor:**

Each ml of 0.1 M KMnO, = 0.139 gm of FeSO, $7H_2O$

Calculations:

The percentage purity of given sample of ferrous sulphate is calculated by using t following

$$\% \text{ of ferrous sulphate} = \frac{\text{Volume of KMnO, used x IP factor x 100 x Mp KMnO}}{\text{Weight of ferrous sulphate (gm) x Mm KMnO}}$$

where, M(P) KMnO, is the calculated morality of KMnO, solution.

M(T)KMnO, is the theoretical morality of KMnO4 solution.

$$\% \text{ purity of ferrous sulphate} = \frac{\text{.....ml KMnO, used x 0.139 x 100 x ....}}{1 \times 0.1}$$

% purity of ferrous sulphate..... ml KMnO4 ..... M KMnO4 × 139 = ……..%•

Inference: The percentage purity of given sample of ferrous sulphate is……..%

EXPERIMENT NO. 10 **DATE:**

TO PERFORM ASSAY OF A GIVEN SAMPLE OF FERROUS SULPHATE

Aim: To perform assay of a given sample of ferrous sulphate.

Requirements:

Chemicals: Ferrous sulphate, 0.1 M potassium permanganate solution, 0.1 M oxalic acid solution, dilute sulphuric acid, distilled water.

Apparatus: Burette, pipette (10 ml), conical flask, beaker, volumetric flask (1000 ml),funnel, glass rod.

Theory: Ferrous sulphate is an example of hematinic. It is a reducing agent and this titration is an example of redox titration (Permanganametry). Potassium permanganate is a powerful oxidizing agent in the presence of ferric sulphate. As soon as oxidation of ferrous sulphate is completed, addition of a drop of KMnO4 produces a permanent pink colour which indicates the end point. The reaction involved in this titration is as follow.

$$2KMnO4 + 10FeSO4 + 2H_2SO \rightarrow K_2SO4 + 2MnSO4 + 8H_2O + 5(Fe_2SO4)$$

Procedure:

Standardisation of KMnO4:

1. Preparation of 0.1 M potassium permanganate solution: Prepare 0.1 M solution by dissolving 15.8 gm of potassium permanganate in 900 ml of water and heat it on a water-bath for 1 hour. Then cool and filter through a sintered glass filter and add sufficient water to produce 1000 ml solution. Store and protect from moisture and sun light.

2. Preparation of 0.1 M oxalic acid solution: Weigh accurately 12.6 gm of oxalicacid and put it into a beaker (250 ml). Add approximately 150 ml of distilledwater and stir it to make clear solution. Transfer this solution to a volumetric flask(1000 ml) and make up the volume. Shake well to mix properly.

3. Titration: Rinse and fill the burette with 0.1 M potassium permanganate solution with the help of funnel. Pipette out 10 ml of 0.1 M oxalic acid solution into a clean and dry conical flask and add 10 ml of dilute sulphuric acid. Shake it to mix the contents. Heat the contents of conical flask to 60-70°C. Titrate the contents of conical flask with 0.1 M potassium permanganate solution until permanent faint pink colour is obtained. Repeat the titration and take three

concordant readings. Calculate molarity of 0.1 M potassium permanganate solution and note it down as MP KMnO4

4. Assay of ferrous sulphate: Weigh accurately about 1 gm of ferrous sulphate and dissolve in 20 ml of dilute sulphuric acid in a conical flask. Titrate the contents of the flask with 0.1 M potassium permanganate until a permanent pink colour is obtained. 1 ml of 0.1 M potassium permanganate is equivalent to 0 139 gm of FeSO, Repeat the titration for 3 times to get concordant values.

- **Result:** The percentage purity of given sample of ferrous sulphate is…….%

• Assay of calcium gluconate:

Sr.no	Volume of calcium gluconate solution (ml)	Burette reading (ml) Initial	Burette reading (ml) Final	Volume of EDTA used (ml)
1	50			
2	50			
3	50			

Average volume of EDTA used =……..ml

• IP factor or equivalent factor:

Each ml of 0.0.5 M EDTA = 0.02242 gm of $C_{12}H_{22}CaO_{14}H_2O$

Calculations:

The percentage purity of given sample of calcium gluconate is calculated by using t following

$$\% \text{ of calcium gluconate} = \frac{\text{Volume of EDTA used x IP factor x 100 x Mp EDTA}}{\text{Weight of calcium gluconate (gm) x Mt EDTA}}$$

where, M(P) EDTA, is the calculated morality of EDTA, solution.

M(T)EDTA, is the theoretical morality of EDTA solution.

$$\% \text{ purity of calcium gluconate} = \frac{\text{.....ml EDTA used x 0.022x 100 x ....M EDTA}}{0.5 \times 0.05}$$

% purity of calcium gluconate..... ml EDTA ..... M EDTA × 88 = ……..%•

Inference: The percentage purity of given sample of calcium gluconate is……..%

EXPERIMENT NO. 11 **DATE:**

TO PERFORM ASSAY OF A GIVEN SAMPLE OF CALCIUM GLUCONATE

Aim: To perform assay of a given sample of calcium gluconate

Requirements:

Chemicals: Calcium gluconate, 0.05 M EDTA solution, 0.05 M calcium chloride solution, 0.05 M magnesium sulphate solution, eriochrome black-T indicator, ammonia buffer (pH 10), distilled water, ammonium chloride, ammonia.

Apparatus: Burette, pipette (10 ml), conical flask, beaker, volumetric flask (1000 ml)..funnel, glass rod.

Theory: Calcium gluconate is calcium D-gluconate monohydrate. Calcium gluconate contains not less than 98.5% and not more than 102.0% of CH2CaO H2O. Its molecular weight is 448.4 gm/mol. It exists as a white, crystalline powder or granules. Calcium gluconate is a mineral supplement and used in treatment of hypokalemia, hypocalcaemia, and magnesium sulphate overdose. This titration is an example of complex metric titration. Complex metric titrations are the reactions in which a metal ion is converted into a stable complex ion by addition of reagent or a ligand. The indicator used is eriochrome black-T indicator which gives blue colour as end point. The following reaction takes place during assay of calcium glaciate:

$$C12H22CaO4 + Na_2H_2Y + 2NH_4 \Rightarrow \quad CaNa_2Y + (NH4)2\ C_{12}H_2CaO14\text{-}H_2O$$

Procedure:

Standardization of EDTA:

1. Preparation of 0.05 M disodium edetate solution: Weigh accurately 18.6 gm of disodium edetate and dissolve it in approximately 900 ml of water. Transfer this solution to a volumetric flask (1000 ml) and make up the volume. Shake it well to mix properly.

2. Preparation of 0.05 M calcium chloride solution: Weigh accurately 14.7 gm of calcium chloride and dissolve it in approximately 900 ml of water. Transfer this solution to volumetric flask (1000 ml) and make up the volume. Shake it well to mix properly.

3. Preparation of ammonia buffer (pH 10): Dissolve 5.4 gm of ammonium chloride in 20 ml of water, add 35 ml of 10 M ammonia and dilute it with water to 100 ml.

4. Titration: Rinse and fill the burette with 0.05 M EDTA solution with the help of funnel. Pipette out 10 ml of 0.05 M calcium chloride solution into a clean and dry conical flask and add 2-3 drops of Eriochrome black T indicator to it. Shake it to mix the contents. Titrate the contents of conical flask 0.05 M EDTA solution until colour is changed from red to blue. Repeat the titration and take three concordant readings Calculate molarity of 0.05 M EDTA solution and note it down as M EDTA.

5. Assay:

i. Preparation of 0.05 M magnesium sulphate solution: Dissolve 12.5 gm of magnesium sulphate in sufficient water to produce 1000 ml.

ii. Assay of calcium gluconate: Weigh accurately about 0.5 gm of calcium gluconate and dissolve it in 50 ml of warm water. Cool and add 5 ml of 0.05 M magnesium sulphate and 10 ml of strong ammonia solution. Titrate with 0.05 M disodium edetate using eriochrome black-T as indicator. Subtract the volume of the magnesium sulphate solution added from the volume of 0.05 M disodium edetate. 1 ml of the remainder of 0.05 M disodium edetate is equivalent to 0.02242 gm of $CHCaOH_2O$. Repeat the titration for 3 times to get concordant values.

Result: The percentage purity of given sample of calcium gluconate is……..%

• **Assay of sodium chloride:**

Sr.no	**Volume of sodium chloride solution (ml)**	**Burette reading (ml) Initial**	**Burette reading (ml) Final**	**Volume of silver nitrate used (ml)**
1	10			
2	10			
3	10			

Average volume of silver nitrate used =……..ml

Calculations:

The percentage purity of given sample of sodium chloride is calculated by using t following

$$\% \text{ of sodium chloride} = \frac{\text{Volume of } AgNO_3 \text{ used} \times \text{Normality of } AgNO_3 \times \text{IP factor} \times 100}{\text{Weight of sodium chloride (gm)} \times \text{normality of } AgNO_3}$$

$$\% \text{ purity of sodium chloride} = \frac{\text{.....ml } AgNO_3 \text{ used} \times 0.00584 \times 100 \times \text{....N} AgNO_3}{0.25 \times 0.1}$$

% purity of sodium chloride..... ml $AgNO_3$..... N $AgN0_3 \times 24$ = ……..%

• **Inference:** The percentage purity of given sample of sodium chloride is……..%

EXPERIMENT NO. 12 **DATE:**

TO PERFORM THE ASSAY OF SODIUM CHLORIDE BY MODIFIED VOLHARD'S METHOD

Aim: To perform the assay of sodium chloride by modified Volhard's method

Requirements:

Chemicals: Sodium Chloride, silver nitrate, nitric acid, potassium chromate indicator, distilled water.

Apparatus: Burette, pipette (10 ml), conical flask, beaker, volumetric flask (1000 ml), funnel glass rod,

Theory: Sodium chloride is also known as common salt. Sodium chloride contains not less than 99.5% and not more than 100.5% of NaCl. It is a white crystalline powder or colourless crystals, odourless and has saline taste. It is freely soluble in water slightly more in boiling water, soluble in glycerine. Sodium chloride is used as an electrolyte replenishes emetic and used in homeopathic medicine Solution of sodium chloride may be used as an eye drop, nasal drop and as a mouthwash. This titration is an example of Volhard's precipitation method titration. Volhard's method is an indirect or back titration method in which an excess of a standard solution of silver nitrate is added to a chloride containing sample solution. The indicator used is potassium chromate indicator which gives reddish brown colour as end point. The following reaction takes place during assay of sodium chloride:

$$NaCl + AgNO_3 \longrightarrow AgCl + NaNO_3$$

$$K_2CrO_4 + 2\,AgNO_3 \longrightarrow Ag,Cro_2 + 2\,KNO;$$

Procedure:

1. Preparation of 0.1 N silver nitrate solutions: Weigh accurately 16.99 gm of silver nitrate and dissolve it in approximately 900 ml of distilled water. Transfer this solution to a volumetric flask (1000 ml) and make up the volume. Shake it well to mix properly.

2. Preparation of 0.1 N sodium chloride solutions: Weigh accurately 5.84 gm of sodium chloride and dissolve it in approximately 900 ml of distilled water. Transfer this solution to volumetric flask (1000 ml) and make up the volume. Shake it well to mix properly.

3. Preparation of 5% potassium chromate indicator: Dissolve 5 gm of potassium chromate in 100 ml of distilled water. 4. Titration: Rinse and fill the burette with 0.1 N silver nitrate solution with the help of funnel. Pipette out 10 ml of 0.1 N sodium chloride solution into a clean

and dry conical flask and add 2-3 drops of potassium chromate indicator to it. Shake it to mix the contents. Titrate the contents of conical flask 0.1 N silver nitrate solution until colour is changed from reddish brown. Repeat the titration and take three concordant readings. Calculate normality of 0.1 N silver nitrate solution.

5. Assay: Assay of sodium chloride: Weigh accurately about 0.25 gm of sodium chloride and dissolve it in 50 ml of distilled water. Add 1 ml of potassium chromate indicator in it. Titrate with 0.1 N silver nitrate solution till the appearance of reddish-brown precipitate. Repeat the titration for 3 times to get concordant values.

Result: The percentage purity of given sample of sodium chloride is…….%

• **Assay of ascorbic acid:**

Sr.no	**Volume of ascorbic acid solution (ml)**	**Burette reading (ml) Initial**	**Burette reading (ml) Final**	**Volume of iodine used (ml)**
1	10			
2	10			
3	10			

Average volume of iodine used =……..ml

Calculations:

The percentage purity of given sample of ascorbic acid is calculated by using t following

$$\% \text{ of ascorbic acid} = \frac{\text{Volume of iodine used x Normality of iodine x IP factor x 100}}{\text{Weight of ascorbic acid (gm) x normality of iodine}}$$

$$\% \text{ purity of ascorbic acid} = \frac{\text{.....ml iodine used x 0.008806 x 100 x ....M iodine}}{0.25 \times 0.1}$$

% purity of ascorbic acid..... ml iodine..... M iodine × 36 = ……..%

• **Inference:** The percentage purity of given sample of ascorbic acid is……..%

EXPERIMENT NO. 13 **DATE:**

TO PERFORM THE ASSAY OF THE ASCORBIC ACID BY CERIMETRY

Aim: To perform the assay of the ascorbic acid by cerimetry.

Requirements:

Chemicals: Vitamin C tablets, iodine, starch indicator, distilled water.

Apparatus: Burette, pipette (10 ml), conical flask, beaker, volumetric flask (1000 ml). funnel, glass rod. **Theory:** Ascorbic acid (Vitamin C) is a water soluble vitamin. It is a vitamin found in various foods and sold as a dietary supplement. It occurs as a white or slightly yellow crystal or powder with a slight acidic taste. Ascorbic acid is freely soluble in water. sparingly soluble in alcohol, insoluble in chloroform, in ether and in benzene. The chemical name of ascorbic acid is L-ascorbic acid. It is used to prevent and treat scurvy. It is a mild reducing agent. Cerimetry is a method of volumetric chemical analysis

Procedure:1. Preparation of 0.1 M iodine solution: Weigh accurately 10 gm of potassium iodide and take in a 250 ml volumetric flask and add 35 ml of distilled water followed by heating the solution, cool the mixture to room temperature and dissolve 3.15 gm of solid iodine powder. Shake it well to mix properly.

2. Preparation of vitamin C solution: Weigh accurately 25 mg of ascorbic acid and take in a 100 ml beaker and dissolve in 100 ml distilled water, Shake it well to mix properly.

3. Preparation of starch indicator. Addition of 0.25 gm of starch powder in 50 ml warm distilled water.

4. Titration: Rinse and fill the burette with 0.1 M iodine solution with the help of funnel. Pipette out 10 ml of vitamin C solution into a clean and dry conical flask and add 2-3 drops of starch indicator to it. Shake it to mix the contents. Titrate the contents of conical flask 0.1 M iodine solution until colour is changed from dark blue to black colour. Repeat the titration and take three concordant readings. Calculate molarity of 0.1 M iodine solution.

5. Assay:

Assay of ascorbic acid: Weigh accurately about 0.25 gm ascorbic acid and dissolve it in 50 ml of distilled water. Add 2-3 drops of starch indicator in it. Titrate with 0.1 M iodine solution till the appearance of dark blue to black precipitate. Repeat the titration for 3 times to get concordant values.

Result: The percentage purity of given sample of ascorbic acid is…… %.

• **Assay of ibuprofen:**

Sr.no	**Volume of ibuprofen solution (ml)**	**Burette reading (ml) Initial**	**Burette reading (ml) Final**	**Volume of NAOH used (ml)**
1	10			
2	10			
3	10			

Average volume of NAOH used =……..ml

Calculations:

The percentage purity of given sample of ibuprofen is calculated by using t following

$$\% \text{ of ibuprofen} = \frac{\text{Volume of NAOH used x Normality of NAOH x IP factor x 100}}{\text{Weight of ibuprofen (gm) x normality of NAOH}}$$

$$\% \text{ purity of ibuprofen} = \frac{\text{.....ml NAOH used x 0.02042x 100 x ....M NAOH}}{0.4 \times 0.1}$$

% purity of ibuprofen..... ml NAOH..... M NAOH × 51= ……..%

• **Inference:** The percentage purity of given sample of ibuprofen is……..%

EXPERIMENT NO. 14 **DATE:**

TO PERFORM THE ASSAY OF THE IBUPROFEN BY ALKALIMETRY

Aim: To perform the assay of the ibuprofen by alkalimetry

Requirements:

Chemicals: Ibuprofen, Sodium hydroxide, ethanol, phenolphthalein indicator solution, distilled water.

Apparatus: Burette, pipette (10 ml), conical flask, beaker, volumetric flask (1000 ml), funnel, glass rod.

Theory: Ibuprofen is an iso-butyl propanoic phenolic acid. Ibuprofen is a white, crystalline, weakly acidic solid substance with slight odour. Ibuprofen is more soluble in alcohols, very slightly soluble in water. Ibuprofen reduces inflammation. It reduces fever and treats pain. The assay of Ibuprofen is carried out by aqueous acid base titration. It is weak acid with strong base type of neutralization titration. It is based on neutralization reaction of Ibuprofen with sodium hydroxide using phenolphthalein solution as an indicator.

Procedure:

1. Preparation of 0.1 M Sodium hydroxide solution: Weigh accurately 4.0 gm of sodium hydroxide and dissolve it in approximately 900 ml of distilled water. Transfer this solution to a volumetric flask (1000 ml) and make up the volume. Shake it well to mix properly.

2. Phenolphthalein solution (indicator): Weigh out 0.5 gm of phenolphthalein. Prepare a 50% ethanol (ethyl alcohol) solution consisting of 50 ml ethanol and 50 ml water. Dissolve the phenolphthalein thoroughly in the 50% ethanol solution. Use from a bottle fitted with an eye dropper. Store the rest in a stoppered bottle.

3. Titration: Rinse and fill the burette with 0.1 M sodium hydroxide solution with the help of funnel. Pipette out 10 ml of ibuprofen solution into a clean and dry conical flask and add 2-3 drops of phenolphthalein indicator to it. Shake it to mix the contents. Titrate the contents of conical flask 0.1 M sodium hydroxide solution until colour is changed from permanent faint pink colour. Repeat the titration and take three concordant readings. Calculate molarity of 0.1 M sodium hydroxide solution.

Assay of ibuprofen: Weigh accurately about 0.4 gm of ibuprofen and dissolve 4. Assay: in 100 ml of ethanol. Add 0.2 ml of phenolphthalein solution indicator in it. Titrate with 0.1 M sodium

hydroxide solution till the appearance of permanent faint p colour. Repeat the titration for 3 times to get concordant values. pink

Result: The percentage purity of given sample of ibuprofen is……..%

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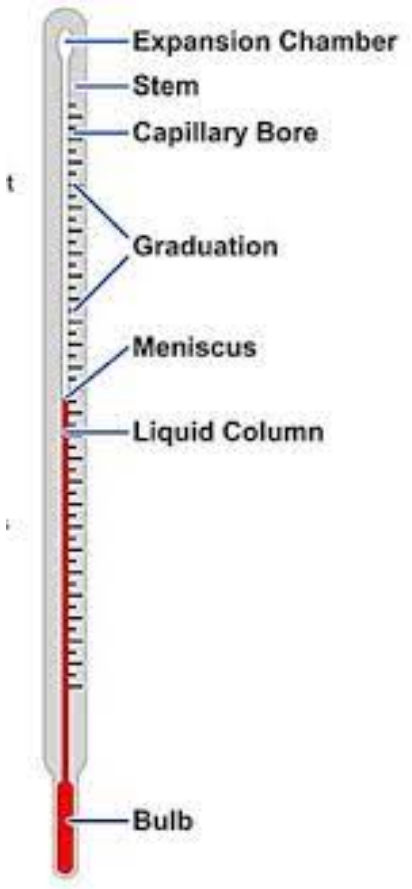


Melting Point Apparatus **Capillary With Thermometre**

Observation:

Test	Temparature noted degree
I	
II	
III	

The total point of naphthalene =<u>T1 +T2+T3</u>

3

=…….C

EXPERIMENT NO. 15 **DATE:**

TO DETERMINE THE MELTING POINT OF ORGANIC COMPOUNDS LIKE NAPHTHALENE

Aim: To determine the melting point of organic compounds like naphthalene

Requirements:

Chemical: Naphthalene,

Apparatus: The aluminium block, stand with clamp, capillary tube, tripod. Thermometer, and kerosene burner.

Theory:

Melting point is a characteristic property of solid crystalline substances. It is the temperature at which the solid phase changes to the liquid phase. Melting point determination is the thermal analysis most frequently used to characterize solid crystalline materials. It is used in research and development as well as in quality control in various industry segments to identify solid crystalline substances and to check their purity.

Procedure:1. Take a capillary tube and close its one end by heating the end in the flame for 2-3 minutes while continuously rotating it.

2. Take naphthalene on a tile and crush it into a fine powder.

3. As shown in the figure above, firmly hold the closed end of the capillary tube between your fingers and thumb.

4. Dip the open end of the capillary tube in the finely powdered naphthalene.

5. Gently tap the capillary tube on the table to fill the compound in the capillary tube to about a length of 1-2 cm.

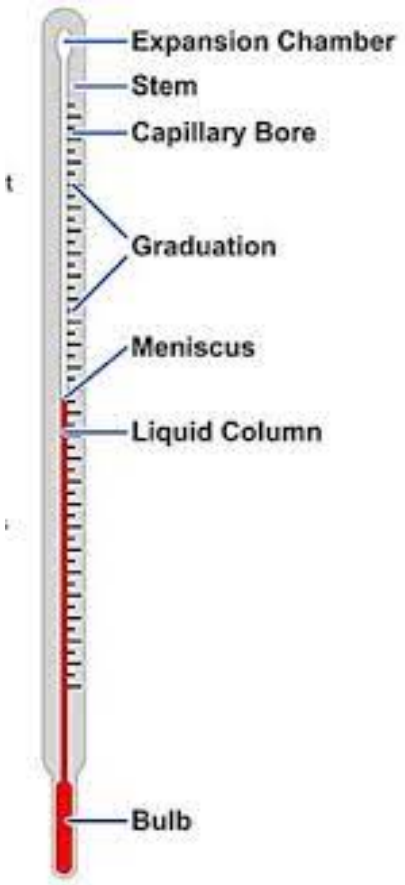
6. With the help of a thread, attach the capillary tube to a thermometer as shown in the figure.

7. Place the capillary tube in the groove of the aluminium block.

8. Double check that the capillary tube holding the naphthalene is in the middle of the groove.

9. Place the Aluminium block on a tripod stand above the kerosene burner and start heating the block with the burner

.**Result:** The melting point of Naphthalene is…….



Melting Point Apparatus **Capillary With Thermometre**

Observation:

Test	Temparature noted degree
I	
II	
III	

The total point of naphthalene =T1 +T2+T3

3

=…….C

EXPERIMENT NO. 16 **DATE:**

TO DETERMINE THE MELTING POINT OF ORGANIC COMPOUNDS LIKE BENZOIC ACID

Aim: To determine the melting point of organic compounds like benzoic acid,

Requirements:

Chemicals: Benzoic acid.

Apparatus: The aluminium block, stand with clamp, capillary tube, tripod,thermometer, and kerosene burner.

Theory: Melting point is a characteristic property of solid crystalline substances. It is the temperature at which the solid phase changes to the liquid phase. Melting point determination is the thermal analysis most frequently used to characterize solid crystalline materials. It is used in research and development as well as in quality control in various industry segments to identify solid crystalline substances and to check their purity

Procedure:1. Take a capillary tube and close its one end by heating the end in the flame for 2 3 minutes while continuously rotating it.

2. Take naphthalene on a tile and crush it into a fine powder.

3. As shown in the figure above, firmly hold the closed end of the capillary tube between your fingers and thumb.

4. Dip the open end of the capillary tube in the finely powdered naphthalene.

5. Gently tap the capillary tube on the table to fill the compound in the capillary tube to about a length of 1-2 cm.

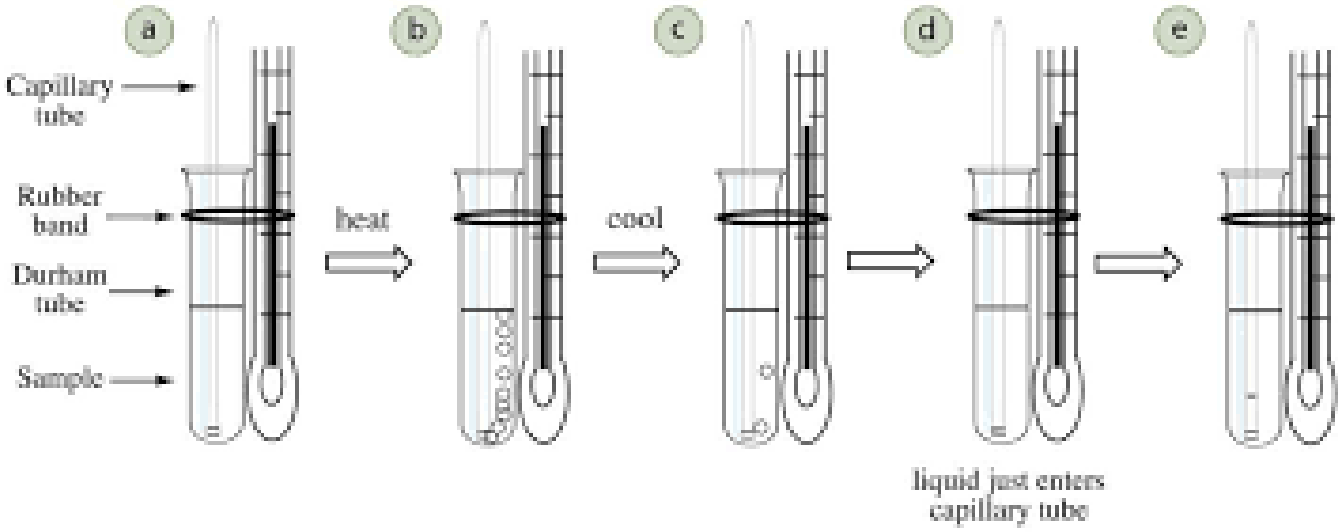
6. With the help of a thread, attach the capillary tube to a thermometer as shown in the figure.

7. Place the capillary tube in the groove of the aluminium block.

8. Double check that the capillary tube holding the naphthalene is in the middle of the groove.
9.Place the Aluminium block on a tripod stand above the kerosene burner and start heating the block with the burner.

10. Keep continuous watch of the temperature and note the temperature as soon as

Result: The melting point of Benzoic acid is……



Determination of boiling point

Boiling Point Apparatus

EXPERIMENT NO. 17 **DATE:**

TO DETERMINE THE BOILING POINT OF ORGANIC UNKNOWN COMPOUNDS

Aim: To determine the boiling point of organic unknown compounds

Requirements

Chemicals: Pure given organic compound, liquid paraffin. Apparatus: Thiele's tube, capillary tube, Stand, thread, Bunsen burner.

Theory: Different methods are used for the determination of boiling point including; distillation, reflux and by using Thiele tube. The most important method uses a Thiele tube and has an advantage of using less than 0.5 ml of material. Thiele Tube Method: The Thiele tube method is one of the simplest methods to determine a compound's boiling point, and has the advantage of using small amounts of material (less than 0.5 ml of sample). The sample is placed in a small tube along with an inverted capillary tube. The setup is attached to a thermometer and heated inside a Thiele tube to slightly higher than the compound's boiling point (which is evidenced by a continuous stream of bubbles emerging from the capillary tube). The tube is then allowed to cool, and the moment liquid is drawn into the capillary tube, the temperature is the compound's boiling point.

This method utilizes the definition of boiling point: The temperature where the compound's vapour pressure equals the applied (atmospheric) pressure. The inverted capillary tube acts as a reservoir to trap the compound's vapours. As the apparatus is heated, the air initially trapped in the capillary tube expands and causes bubbles to emerge from the tube. With further heating, the compound's vapours eventually displace all of the trapped air, which is why heat is applied until there is a continuous stream of bubbles.When the apparatus is cooled, eventually the pressure inside the capillary tube (due solely to the compound's vapours) will match the atmospheric pressure, at which point the bubbles will slow and liquid will be drawn into the tube. The temperature where this begins is the compound's boiling point.

• **Procedure**:

1. Obtain a Thiele tube and clamp it to a ring stand in the fume hood. The tube is normally filled with clear mineral oil, but it may have darkened from oxidation or spilled compounds. If the oil is quite dark, it should be replaced. The oil should be filled to at least 1 cm higher than the top triangular arm (an appropriate oil level), and if too low the oil will not circulate as needed.

Observation:

Sr.no	Name of the compound	Boiling point in degree celsius
1	Acetic acid (known compound)	118
2	Unknown compound	

Boiling point of some organic compound:

Sr.no	Name of the compound	Boiling point in degree celsius
1	Nitrobenzene	210
2	Benzene	80
3	Benzaldehyde	179
4	Acetone	56

2.insert a thermometer into a one-holed rubber stopper with a slit down one side. Attach a small glass vial ("Durham tube, or 66 50 mm culture tube) to the thermometer with a small rubber band. The bottom of the vial should be flush with the bottom of the thermometer.

3. Fill the vial about half-full with sample, which will require between 0.25-0.5 ml ofsample.

4. I nsert a capillary tube into the sample (the same type that is used for meltingpoints), open end down and sealed end up. Place the rubber stopper andthermometer assembly into the Thiele tube, adjusting the height so that thesample is midway (if possible) inside the tube. The rubber band should be higher than the top of the mineral oil keeping in mind that the oil may expand somewhat during heating. The thermometer should not touch the sides of the glass, and if it does it should be clamped in such a way that it no longer touches.

5. Heat the oil gently on the side arm of the Thiele tube with a microburner if available, or Bunsen burner using a back-and-forth motion As the oil warms and becomes less dense, it will rise and travel up the triangular portion of the tube. The cooler, denser oil will sink, thereby creating a current. This method is an excellent way to indirectly and slowly heat the sample.

6. Although bubbles should not be seen in the Thiele tube as it warms, they commonly are seen if the tube had been used previously for boiling point determinations. In this method, the rubber band occasionally breaks causing the sample to fall into the oil and contaminate it. If the oil is not subsequently changed, the sample may boil when heated in the tube. It is okay to continue heating a Thiele tube if bubbles are seen.

7. It is best to heat the oil gently and in a continual manner, as stopping andstarting have caused the results to suffer.

8. Continue heating until a vigorous stream of bubbles emerges from the tip of the capillary tube, such that individual bubbles can barely be distinguished. The purpose of this step is to expunge the air originally present in the capillary tube and replace it with the sample's vapour. Do not heat so vigorously that the entire sample boils away. When bubbles are vigorously emerging from the capillary tube, the vapour pressure inside the tube is greater than the atmospheric pressure (the oil is at a higher temperature than the boiling point).

9. Turn off the burner and allow the apparatus to cool. The bubbles will slow and eventually stop. At some point the vapour pressure inside the capillary tube will equal the atmospheric pressure and liquid will be drawn into the tube. The boiling point should be recorded as the temperature when liquid just begins to enter the capillary tube.

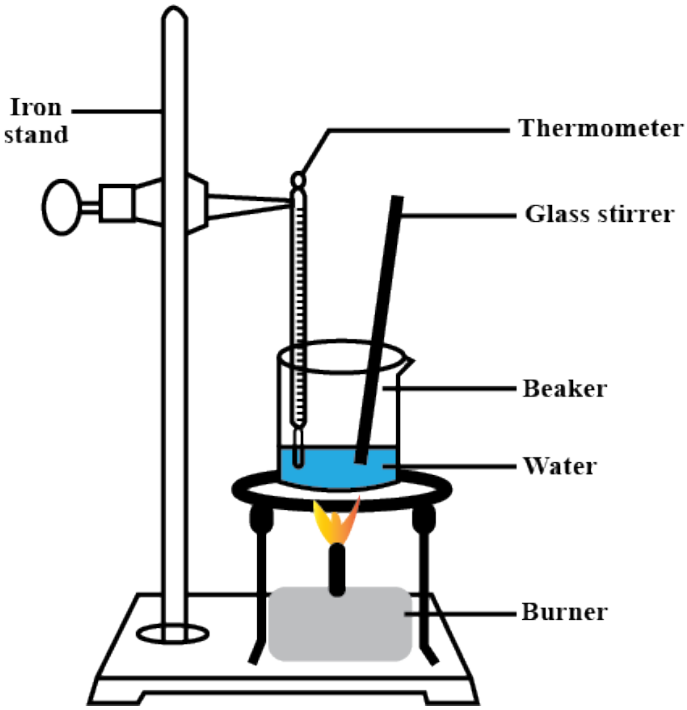
10. Record the atmospheric pressure along with the boiling point.

Result:

1) The Boiling point of given organic Liquid……..._is…….

2) The Boiling point of given unknown organic liquid is…….

3) The Unknown organic liquid is_.....



Determine The Boiling Point Of Water

boiling point apparatus

EXPERIMENT NO. 18 **DATE:**

TO DETERMINE THE BOILING POINT OF BENZENE

Aim: To determine the boiling point of benzene

Requirements:

Chemicals: Pure given organic compound, liquid paraffin, benzene

Apparatus: Thiele's tube, capillary tube, stand, thread, bunsen burner Theory:

Different methods are used for the determination of boiling point including distillation, reflux and by using Thiele tube. The most important method uses a Thiele tube and has an advantage of using less than 0.5 ml of material.

Thiele Tube Method: The Thiele tube method is one of the simplest methods to determine a compound's boiling point, and has the advantage of using small amounts of material (less than 0.5 ml of sample). The sample is placed in a small tube along with an inverted capillary tube. The setup is attached to a thermometer and heated inside a Thiele tube to slightly higher than the compound's boiling point (which is evidenced by a continuous stream of bubbles emerging from the capillary tube). The tube is then allowed to cool, and the moment liquid is drawn into the capillary tube, the temperature is the compound's boiling point.This method utilizes the definition of boiling point: the temperature where the compound's vapour pressure equals the applied (atmospheric) pressure. The inverted capillary tube acts as a reservoir to trap the compound's vapours. As the apparatus is heated, the air initially trapped in the capillary tube expands and causes bubbles to emerge from the tube. With further heating, the compound's vapours eventually displace all of the trapped air, which is why heat is applied until there is a continuous stream of bubbles.When the apparatus is cooled, eventually the pressure inside the capillary tube (due solely to the compound's vapours) will match the atmospheric pressure, at which point the bubbles will slow and liquid will be drawn into the tube. The temperature where this begins is the compound's boiling point.

Procedure:

1. Obtain a Thiele tube and clamp it to a ring stand in the fume hood. The tube is normally filled with clear mineral oil, but it may have darkened from oxidation or spilled compounds. If the oil is quite dark, it should be replaced. The oil should be filled to at least 1cm1cm higher than the top triangular arm (an appropriate oil level), and if too low the oil will not circulate as needed.

Observation:

Sr.no	Name of the compound	Boiling point in degree celsius
1	Acetic acid (known compound)	118
2	Unknown compound	

2 Insert a thermometer into a one-holed rubber stopper with a slit down one side. Attach a small glass vial ("Durham tube", or 66 x 50 mm culture tube) to the thermometer with a small rubber band. The bottom of the vial should be flush with the bottom of the thermometer.

3. Fill the vial about half-full with sample, which will require between 0.25-0.5 ml of sample.

4. Insert a capillary tube into the sample (the same type that is used for melting points), open end down and sealed end up. Place the rubber stopper and thermometer assembly into the Thiele tube, adjusting the height so that the sample is midway (if possible) inside the tube. The rubber band should be higher than the top of the mineral oil keeping in mind that the oil may expand somewhat during heating. The thermometer should not touch the sides of the glass, and if it does it should be clamped in such a way that it no longer touches.

5. Heat the oil gently on the side arm of the Thiele tube with a microburner if available, or Bunsen burner using a back-and-forth motion As the oil warms and becomes less dense, it will rise and travel up the triangular portion of the tube. The cooler, denser oil will sink, thereby creating a current. This method is an excellent way to indirectly and slowly heat the sample.

6. Although bubbles should not be seen in the Thiele tube as it warms, they commonly are seen if the tube had been used previously for boiling point determinations. In this method, the rubber band occasionally breaks causing the sample to fall into the oil and contaminate it. If the oil is not subsequently changed, the sample may boil when heated in the tube. It is okay to continue heating a Thiele tube if bubbles are seen.

7. It is best to heat the oil gently and in a continual manner, as stopping and starting have caused the results to suffer.

8. Continue heating until a vigorous stream of bubbles emerges from the tip of the capillary tube, such that individual bubbles can barely be distinguished. The purpose of this step is to expunge the air originally present in the capillary tube and replace it with the sample's vapour. Do not heat so vigorously that the entire sample boils away. When bubbles are vigorously emerging from the capillary tube, the vapour pressure inside the tube is greater than the atmospheric pressure (the oil is at a higher temperature than the boiling point).

9. Turn off the burner and allow the apparatus to cool. The bubbles will slow and eventually stop. At some point the vapour pressure inside the capillary tube will equal the atmospheric pressure and liquid will be drawn into the tube. The boiling point should be recorded as the temperature when liquid just begins to enter the capillary tube

10. Record the atmospheric pressure along with the boiling point.

• **Result:**

1) The Boiling point of given organic Liquid ……..is……°C.

2) The Boiling point of benzene is……. °C.

Calculation:

The crude yield of Picric acid was calculated as follows:

Molecular weight of phenol = 94.11 gm/mol

Molecular formula of picric acid = $CH,NO,$

Molecular formula of phenol = CHO

Molecular weight of picric acid = 229.1 gm/mol

94.11g of phenol forms 229.1 gm of picric acid

4 gm phenol will form x gm of picric acid

$X = 229.1 \times 4/94.11 = 9.8$ gm

Theoretical yield = 9.8 gm

Experimental yield = ___gm

Inference: The percentage yield of crude Picric acid is……

EXPERIMENT NO. 19 **DATE:**

TO PREPARE PICRIC ACID FROM PHENOL.

Aim: To prepare picric acid from phenol.

Requirements:

Chemicals: Phenol, Sulphuric acid, Nitric acid, Nitrobenzene etc.

Apparatus: Conical flask, beaker, measuring cylinder, volumetric flask etc.

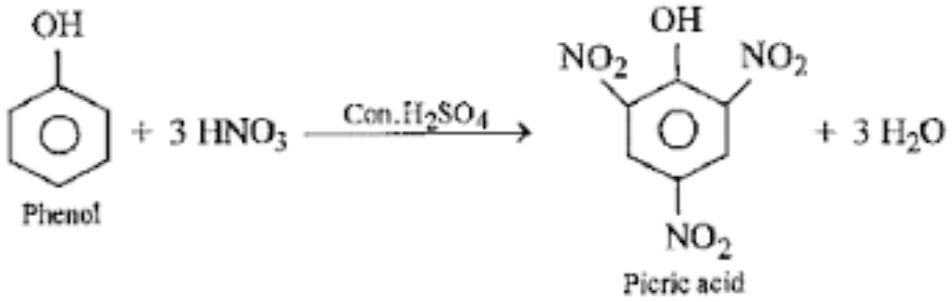
Theory:

Picric acid is 2,4,6-trinitrophenol. It is yellow in colour. It is used as a disinfectant. It is obtained by nitrating phenol. Nitration: Nitration is an example of electrophilic aromatic substitution reaction. A large number of aromatic compounds can be easily nitrated. The hydrogen atom replaced by nitro group. The nitration of aromatic compounds is usually done by using conc nitric acid in presence of conc. sulphuric acid. Sulphuric acid not only provides strong acidic medium but it also converts the nitric acid into reactive electrophilic nitronium ion which attacks the aromatic ring. Nitration is usually carried out at low temperature. At high temperature there is a loss of material due to oxidation of nitric acid. Phenol undergoes nitration with nitric acid even at room temperature forming ortho and para nitrophenol which can be separated by steam distillation.

Procedure:

Take 4 g of phenol into a dry 250 ml of conical flask. Add 5 ml of conc. sulphuric acid and mix thoroughly which becomes warm because the reaction is exothermic. Now heat the flask on boiling water bath for 30 min. to complete the formation of phenol Sulphuric acid and chill the flask in ice water mixture. Place the flask on a wooden block add 15 ml of conc. Nitric acid and at once mix the liquid by shaking for few minutes. Then allow the mixture to stand undisturbed. Usually within one minute a vigorous but harmless reaction occurs and red fumes pour out of the flask when the action subsides, heat the flask on boiling water bath for 1-2 hours with occasional stirring. During this period, the heavy oils which is present at the beginning ultimately forms a mass of crystals. Add 100 ml of cold water and chill thoroughly, mixing well. Filter the yellow crystals wash thoroughly with water to eliminate all organic acids and drain.

Chemical reaction:



Result: The percentage yield of crude Picric acid is …….

Calculation:

Molecular formula of benzamide CH,NO

Molecular formula of benzoic acid = CH6O2

Molecular weight of Benzamide= 121.14 gm/mol

Molecular weight of benzoic acid = 122.12 gm/mol

121.14 gm of benzamide forms122.12 gm of Benzoic acid

3 g Benzamide will form x gm of Benzoic acid

X = 122.12 x 3/121.14 = 3 gm

Theoretical yield = 3 gm

Experimental yield = ... Gm

$$\textbf{Percentage yield} = \frac{\text{Amount of Benzoic Acid} \times 100}{\text{Theoretical yield of Benzoid Acid}}$$

• **Inference:** The percentage yield of crude benzoic acid……

EXPERIMENT NO. 20 **DATE:**

PREPARATION OF BENZOIC ACID FROM BENZAMIDE

Aim: Preparation of benzoic acid from benzamide.

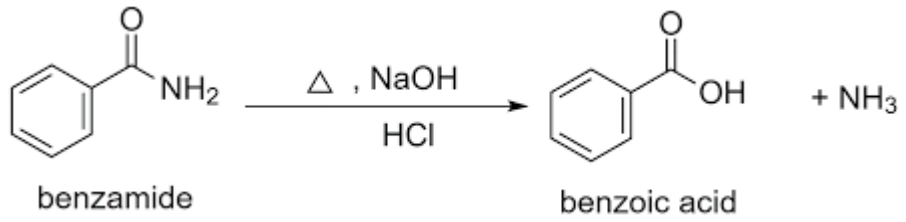
Requirements:

Chemicals: Benzamide, NaOH, HCI.

Apparatus: Round bottom flask, Beaker, Melting Point apparatus, Measuring cylinder, Glass rod, Filter paper, Dropper.

Theory:

Benzamide is a white solid with the chemical formula of CH,C(O)NH, It is the simplest amide derivative of benzoic acid. It is slightly soluble in water and soluble in many organic solvents,



Procedure:

Weight 3 gm of Benzamide and place in a 200 ml RBF. Add 50 ml of 10% sodium hydroxide. Fit the flask with a cork carrying a air condenser and boil the mixture for about 15 minutes. Allow the reaction mixture to cool and transfer to a 250 ml beaker. Add excess dilute hydrochloric acid with dropper. Swirl the container continuously until a precipitate obtained. Filter the solution under suction. Wash the crystals thoroughly with minimum amountof ice cold water. The crude solid is recrystallized from boiling. Water and filter toobtain pure crystals of the product.

Result: The percentage yield of crude benzoic acid……

Calculation:

The crude yield of aspirin was calculated as follows:

Percentage yield = $\frac{\text{Amount of Benzoic Acid x 100}}{\text{Theoretical yield of Benzoid Acid}}$

$\frac{\text{…….mg x100}}{\text{……mg}}$ =

Inference: The percentage yield of crude aspirin……...

EXPERIMENT NO. 21 **DATE:**

TO PERFORM IDENTIFICATION AND TEST FOR PURITY OF ASPIRIN.

Aim: To perform identification and test for purity of aspirin.

Requirements:

Chemicals: Sodium hydroxide, dil. Sulphuric acid and methyl alcohol.

Apparatus: Conical flask (100 ml), beaker (250 ml), measuring cylinder (100 ml), glass rod, spatula, funnel.

Theory:

Chemically aspirin is acetyl salicylic acid and is named as 2-acetoxybenzoic acid. It occurs as colourless crystals. It is odourless. Aspirin is slightly soluble in water, freely soluble in alcohol and soluble in chloroform and ether. Aspirin is used as analgesic, anti-inflammatory and antipyretic. It melts at about 120°C,

Procedure:

1. Boil about 0.5 gm of given sample with 10 ml of sodium hydroxide for threeminutes, cool and add 10 ml of dilute sulphuric acid. A white, crystallineprecipitate is produced and the odour of acetic acid is perceptible. Filter, dissolvethe precipitate in water and to the solution add ferric chloride test solution. Adeep violet colour is produced.

2. Boil about 0.5 gm of given sample with 10 ml of sodium hydroxide for five minutes, cool and add 10 ml of dilute sulphuric acid. A white, crystalline precipitate is produced and the odour of acetic acid is perceptible. Filter, and then add 3 ml of alcohol and 3 ml of sulphuric acid to the filtrate and warm. The odour of ethyl acetate is perceptible.

3. Determine the melting point of aspirin with the help of melting point apparatus.

Result: The given sample is found to be of ……..with melting point…….

Observations for Purity of Caffeine:

Sr.no	Experiment	Observation	Inference
1	10 mg sample 1 ml HCI + 01 gm potassium chlorate Evaporate on water bath. Expose the residue to dil, NH, solution.	Purple colour appears	Caffeine o Present o Absent
2	Saturated solution of sample + few drops of tannic acid.	White precipitate	Caffeine o Present o Absent
3	5 ml sample + 1.5 ml of 0.1 N iodine + few drops of dil. HCI.	Brown precipitate	Caffeine o Present o Absent
4	Melting point	235-238°C	M.P. of caffeine……. to ……°C.

Inference:

The given sample is found to be of……with melting point…….

EXPERIMENT NO. 22 **DATE:**

TO PERFORM IDENTIFICATION AND TEST FOR PURITY OF ASPIRIN.

Aim: To perform identification and test for purity of caffeine

Requirements

Chemicals: Hydrochloric acid, potassium chlorate. ammonia, tannic acid, iodine,sodium hydroxide.

Apparatus: Porcelain dish, test tube, beaker (250 ml), measuring cylinder (100 ml). glass rod, spatula, funnel.

Theory: Caffeine is 1, 3, 7-trimethylxanthine or its monohydrate. It forms silky white crystals or white glistering needles or a white crystalline powder. It is odourless. The taste is bitter. It is sparingly soluble in water and alcohol, and it is freely soluble in chloroform and boiling water. The melting point of Caffeine was found to be 235-238 C

Procedure:

1. Take 10 mg of the sample in a porcelain dish. Add 1 ml of hydrochloric acid and 0.1 gm of potassium chlorate, and evaporate to dryness on a water bath. Expose the residue to the vapours of dilute ammonia solution. A purple colour which disappears on the addition of a fixed alkali is produced.

2. To a saturated solution of sample, add a few drops of tannic acid solution. Awhite precipitate is produced which is soluble in excess of the reagent.

3. To 5 ml of a saturated solution of the sample, add 1.5 ml of 0.1 N iodine. The solution remains clear. Add a few drops of dil. Hydrochloric acid. A brown precipitate is formed. On neutralization with sodium hydroxide solution, the precipitate re-dissolves.

4. Measure the melting point of caffeine with the help of melting point apparatus.

Result: The given sample is found to be of……with melting point……

Observations for purity of paracetamol Experiment :

Sr.no	Experiment	Observation	Inference
1	01 gm sample 10 ml distilled water 005 mi ferric chloride solution	Violet colour is produced.	Caffeine o Present o Absent
2	025 gm sample 4 ml pyridine 05 gm of 4-nitrobenzylchloride. Boil for 2-3 minutes andthen cool, Add mixture to 40 ml distilled water and stir.	Precipitate forms.	Caffeine o Present o Absent
3	01 gm sample 1 ml conc. HCI.Boil for 2-3 minutes. No precipitate is formed. Then add 10 ml distilled water and cool. Then add 0.05 ml potassium dichromate solution	Violet colour is produced.	Caffeine o Present o Absent
4	Melting point	169-172°C	M.P. of paracetamol……. to ……°C.

Inference:

The given sample is found to be of……with melting point……

EXPERIMENT NO. 23 **DATE:**

TO PERFORM IDENTIFICATION AND TEST FOR PURITY OF PARACETAMOL

Aim: To perform identification and test for purity of paracetamol.

Requirements:

Chemicals: Ferric chloride, conc. Hydrochloric acid, potassium dichromate, pyridine, nitrobenzyl chloride, water.

Apparatus: Test tube, test tube stand, glass rod, beaker, measuring cylinder, burner and holder.

Theory:

Paracetamol is 4-hydroxyacetanilide. It is white crystalline powder, sparingly solublein water. It is a p-aminophenol derivative and is used as analgesic and antipyretic. It is kept in well closed container to protect from light. The melting point of paracetamol ranges from 169 to 172°C.

Procedure:

1. Dissolve 0.1 gm of sample in 10 ml of water and add 0.05 ml of ferric chloridesolution. A violet colour is produced.

2. Dissolve 0.2 gm of sample in 4 ml of pyridine solution and add 0,5 gm of4-nitrobenzyl chloride solution. Boil for 2-3 minutes. Cool and pour the reactionmixture in 40 ml of water with continuous stirring. Precipitates are formed.

3. Dissolve 0.1 gm of sample in 1 ml of conc. Hydrochloric acid and boil it for few minutes. No precipitates are formed. Then add 10 ml of distilled water and cool the reaction mixture. Add 0.05 ml of potassium dichromate solution. Violet colour is produced.

4. Measure the melting point of paracetamol with the help of melting point apparatus.

Result: The given sample is found to be of……with melting point……...

Observations for Purity of Sulphanilamide:

Sr.no	Experiment	Observation	Inference
1	50 mg sample 2 ml dil HCI+ 2 ml B-naphthol 1 gm sodium acetate	Orange precipitate	Sulphonamide o Present o Absent
2	1 gm sample+ 5 ml alcohol + 1 ml H,SO	ethyl Ethyl acetate odour	Sulphonamide o Present o Absent
3	Take 500 mg sample and heat for few minutes.	Oily liquid with acetamide odour	Sulphonamide o Present o Absent
4	Melting point	162-163°C	M.P. of Sulphonamide……. to ……°C.

Inference:

The given sample is found to be of……with melting point……

EXPERIMENT NO. 24 **DATE:**

TO PERFORM IDENTIFICATION AND TEST FOR PURITY OF SULPHANILAMIDE

Aim: To perform identification and test for purity of sulphanilamide.

Requirements:

Chemicals: Hydrochloric acid, B-naphthol, sodium acetate, ethyl alcohol, sulphuric acid.

Apparatus: Test tube, test tube stand, glass rod, beaker, measuring cylinder, burner and holder.

Theory:

The simplest of the sulphonamides is sulphanilamide. Chemically, it is p-aminobenzene sulphonamide. Sulphonamides are bacteriostatic against a wide range of gram negative organisms. Sulphonamides are white or near white crystallinepowders. They are very slightly soluble in water but their sodium salts are freelysoluble. The melting point of sulphonamide is about 162 to 163°C.

Procedure:

1. Dissolve 50 mg of sample in 2 ml warm dilute hydrochloric acid and pour thesolution in 2 ml B-naphthol containing 1 gm sodium acetate. An orangeprecipitate is formed.

2. Heat the sample with ethyl alcohol and sulphuric acid. Ethyl acetate recognizableby its odour produced.

3. Place 500 mg of sample in a test tube, heat gently until it melts and then boil. Anoily liquid, which has characteristic odour of acetamide, condenses on the wall of the test tube.

4. Dissolve 1 gm of sample in 10 ml of distilled water and add 2 ml of acetic acid. Awhite precipitate is produced. Collect the precipitate, wash with cold water anddry it at 105°C for 4 hours. Determine the melting point of the precipitate.

Result: The given sample is found to be of……with melting point…….

Sr.no	Experiment	Observation	Inference
1	**Preliminary Reactions**	Colourless	Absence of Fe 2+, Fe3+, Ni2+,C o 2+
2	**Appearance**	Green Blue Brown Pink	May be Fe 2+ ,Cu2+ . Ni2+ May be Cu2+ May be Fe2+ May be Co2+
3	**Action of heat**: Take a small amount of the given salt in a dry test tube, heat it gently, then strongly.	A colourless gas with a characteristic pungent odour turning moist red litmus paper blue. Reddish brown vapours turning acidified ferrous sulphate paper brown, are obtained. Substance is white when cold or yellow when hot.	May be NH4+. Salt May be NO3 - May be Zn 2+
4	Bluish green flame Apple green Brick red Crimson red	May be Cu2+ May be Ba 2- May be Ca 2- May be Sr 2+	

EXPERIMENT NO. 25 **DATE:**

TO ANALYZE THE PRESENCE OF ACID RADICALS (ANIONS) IN THE GIVEN MIXTURE.

Aim: To analyze the presence of acid radicals (anions) in the given mixture.

Requirements:

Apparatus: Test tubes, Boiling Tubes, Test tube holder, Test tube stand, Corks, Filter Paper, Delievery Tube.

Chemicals: Sodium nitrate, Calcium sulphate, Sodium Chloride, Nitric acid, Sulphuric acid, Silver nitrate, Potassium dichromate, Lead acetate, Ammonium molybdate,alcohol, barium chloride.

Theory:

The qualitative analysis is an analytical technique which concerned with the identification of acidic part of the inorganic material in the form of a single salt or as mixture of two or more simple salts. When simple salt or a mixture of two or more simple salts dissolved in water, it split up in to two types of charged particles one a carries positive charged called as positive ion or cation or basic radicals while the other carries a negative charge called as negative ion or anion or an acidic radical. The phenomenon of breaking of the salts in to ions in solution is termed as dissociation. For example in silver chloride silver ion is basic radical while chloride ion is acidic radical

$$AgCl \Longrightarrow Ag^{++} CI$$

Procedure:

Preparation of Sodium Carbonate Extract: Take about 25 mg of the given mixture in a semi-micro beaker and add 250 mg of pure sodium carbonate and about 5 ml of distilled water to it. Heat the mixture to boiling for 5 minutes. Centrifuge it. Reject the residue and collect the centrifugate which is known as sodium carbonate extract.

Observation table:

Experiment	Observation	Inference
Dilute H2So4 Test: Take small amount of sodium carbonate extract in semi-micro test tube. Neutralize with dilute HNO, and add dilute H2So4 to it.	Smell of vinegar. Colourless gas with effervescence. Colourless gas with smell of rotten eggs. Colourless gas with effervescence having smell of burning sulphur. Colourless gas having smell of burning S. Light brown gas having pungent smell.	CH,COO o Present o Absent CO2+ o Present o Absent S2+ o Present o Absent SO3+ o Present o Absent S2O3 o Present o Absent NO2- o Present o Absent
Conc. H2So4 Test: Take small amount of sodium carbonate extract in semi-micro test tube. Neutralize with dilute HNO, and add conc. H2So4 to it	Colourless gas with a pungent odour	CL- o Present o Absent
Confirmatory test of CI-: **AgNO3 Test:** Take a small amount of the given salt in a dry test tube, heat it gently, then strongly.	White ppt, soluble in NH4OH is formed.	CL- o Present o Absent

Experiment	Observation	Inference
Confirmatory test of Cl-: Chromyl Chloride Test: Take 10 mg of mixture and add 30 mg of powdered and 0.5 ml of conc K2Cr2O7 and H2SO4 On heating, brownish vapour of CrO,Cl appear, which when passed in water give yellow solution. To this solution, add 2 drops of (CH3COO) 2 Pb.	Yellow ppt. is formed. .	CL- o Present o Absent
Confirmatory Test for NO3 Diphenylamine test: Take small amount of sodium carbonate extract in a semi-micro test tube and add few drops of diphenylamine reagent	Blue colouration	NO3- o Present o Absent
Confirmatory Test for NO3: Ring test: Take 0.5 ml of sodium carbonate extract and add 0.5 ml of freshly prepared saturated solution of FeSO4. Centrifuge it. In the centrifugate, conc. H2SO4is added drop wise by the side of test tube.	A deep brown ring at the junction of two liquid is formed.	NO3- o Present o Absent

Test for other anions:

Test for Phosphate: Take 10 mg of mixture with 5-10 drops of conc. HNO3. Boil it until no fumes are there. Now add 10 drops of hot ammonia solution.	Yellow colouration or ppt. is formed.	PO3- o Present o Absent
Test for Borate:	Green flame is formed.	BO3- o Present o Absent
Test for sulphate: Take small amount of sodium carbonate extract in a semi-micro test tube. Neutralize it with dilute HNO, and 5 drops of BaCl solution is added to it	Formation of white ppt. insoluble in conc. HNO, is obtained.	SO4- o Present o Absent

Result: The acid radicals (anions) present in the given mixture are…….

Experiment	Observation	Inference
Preliminary Reactions	Colourless	Absence of Fe 2+, Fe3+, Ni2+,C o 2+
Appearance	Green Blue Brown Pink	May be Fe 2+ ,Cu2+ . Ni2+ May be Cu2+ May be Fe2+ May be Co2+
Action of heat: Take a small amount of the given salt in a dry test tube, heat it gently, then strongly.	A colourless gas with a characteristic pungent odour turning moist red litmus paper blue. Reddish brown vapours turning acidified ferrous sulphate paper brown, are obtained. Substance is white when cold or yellow when hot.	May be NH4+. Salt May be NO3 - May be Zn 2+
Flame test: To a small amount of given salt taken in watch glass and add a drop of conc. HCI and make a paste. Introduce the paste with a help of glass rod to the base of the non-luminous Bunsen burner.	Bluish green flame Apple green Brick red Crimson red	May be Cu2+ May be Ba 2- May be Ca 2- May be Sr 2+

EXPERIMENT NO. 26 **DATE:**

TO ANALYZE THE PRESENCE OF ACID RADICALS (ANIONS) IN THE GIVEN MIXTURE.

Aim: To analyze the presence of acid radicals (anions) in the given mixture.

Requirements:

Apparatus: Test tubes, Boiling Tubes, Test tube holder, Test tube stand, Corks, Filter Paper, Delievery Tube.

Chemicals: Sodium nitrate, Calcium sulphate, Sodium Chloride, Nitric acid, Sulphuric acid, Silver nitrate, Potassium dichromate, Lead acetate, Ammonium molybdate,alcohol, barium chloride.

Theory:

The qualitative analysis is an analytical technique which concerned with the identification of acidic part of the inorganic material in the form of a single salt or as mixture of two or more simple salts. When simple salt or a mixture of two or more simple salts dissolved in water, it split up in to two types of charged particles one a carries positive charged called as positive ion or cation or basic radicals while the other carries a negative charge called as negative ion or anion or an acidic radical. The phenomenon of breaking of the salts in to ions in solution is termed as dissociation. For example in silver chloride silver ion is basic radical while chloride ion is acidic radical

$$AgCl \Longrightarrow Ag^{++} CI$$

Observation table:

Experiment	Observation	Inference
Dilute H2So4 Test: Take small amount of sodium carbonate extract in semi-micro test tube. Neutralize with dilute HNO, and add dilute H2So4 to it.	Colourless gas with effervescence observed.	CO2+ o Present o Absent
Confirmatory test of CO3-: take small amount of mixture in v-tube .add few drops of lime soda water in it till the bulb tube is filled	Colourless gas formed turn lime water milky	CO2+ o Present o Absent
Take small amount of sodium carbonate extract in semi-micro test tube. Neutralize with dilute HNO, and add conc. H2So4 to it	Smell of vinegar. Colourless gas with smell of rotten eggs. Colourless gas with effervescence having smell of burning sulphur. Colourless gas having smell of burning S. Light brown gas having pungent smell.	CHCOO- o Present o Absent S2- o Present o Absent S02- o Present o Absent S203- o Present o Absent NO2- o Present o Absent

Experiment	Observation	Inference
Conc. H2So4 Test: Take small amount of sodium carbonate extract in semi-micro test tube. Neutralize with dilute HNO, and add conc. H2So4 to it	Odourless gas with pungent odour Reddish brown gas with pungent odou Violet vapour Colour less gas is evolved which burns with a blue flame	CL- o Present o Absent Br- o Present o Absent I- o Present o Absent C2o4- o Present o Absent
Confirmatory Test for Br- Take small amount of sodium carbonate extract in semi-micro test tube. Neutralize with dilute HNO, and add conc. AgNo4 to it	Light yellow ppt	Br- o Present o Absent
Confirmatory Test for NO3 Diphenylamine test: Take small amount of sodium carbonate extract in a semi-micro test tube and add few drops of diphenylamine reagent	Blue colouration	NO3- o Present o Absent

Test for other anions:

Test for Phosphate: Take 10 mg of mixture with 5-10 drops of conc. HNO3. Boil it until no fumes are there. Now add 10 drops of hot ammonia solution.	No Yellow colouration or ppt. is formed.	PO3- o Present o Absent
Test for Borate:	No Green flame is formed.	BO3- o Present o Absent

Test for sulphate: Take small amount of sodium carbonate extract in a semi-micro test tube. Neutralize it with dilute HNO, and 5 drops of BaCl solution is added to it	No white ppt.	SO4- o Present o Absent

Result: The acid radicals (anions) present in the given mixture are…….

Br- (bromide ion), CO3(Carbonate ion), No3(Nitrate ion)