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Experiment No. 1

Aim:- To Prepare a pure sample of Potash alum [$K_2SO_4\ Al_2(SO_4)_3 \cdot 24H_2O$]

Theory:- Potash alum is prepared by dissolving an equimolar mixture of hydrated aluminium sulphate and potassium sulphate in minimum amount of water containing a little sulphuric acid and then subjecting the resulting solution to crystalligation when octahedral crystals of potash alum separate out.

$$K_2SO_4 + Al_2(SO_4)_3\ 18H_2O + 6H_2O \longrightarrow K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 24H_2O$$

Potash alum

Requirements:- Two beakers (250 ml), china dish, funnel, funnel stand glass rod, wash bottle, tripod stand and wire gauge, Potassium sulphate, aluminium sulphate and dil H_2SO_4

Procedure:- Take a 250 ml beaker washed it with water and then transferred 2.5g potassium sulphate crystals to it. Added 20ml of water. Stirred to dissolve the crystals warmed is required

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2) We will take other 250ml beaker wash it with water and then transferred 10g aluminium sulphate crystals to it Added about 20ml of water and 1ml of dil H_2SO_4 to prevent hydrolysis of aluminium sulphate heated for about 5 min

3. Mixed the two solution in a china-dish and placed the china dish on a wire gauge placed over a burner. Stirred the solution with a glass rod. Concentrated the solution till crystallization Point is reached. Placed the dish over a beaker containing cold water.

4. Boon crystals of potash alum is separated and Decant off the mother liquor and washed the crystals with a small quantity of ice cold water

5. Dried the crystals by Placing them between filter paper.

Observations :- Weight of crystals obtained = 12.5g
Expected yields = 12.5g
colour of the crystals = colourless
Shape of the crystals = octahedral

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Experiment No. 2

Aim :- To Prepare a pure sample of the complex potassium trioxalatoferrate (III) $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$

Theory :- The compound can be prepared by treating ferric sulphate with barium oxalate which results in formation of ferric oxalate, this soluble ferric oxalate, in the presence of excess oxalate ion (obtained by reacting ferric oxalate with potassium oxalate) yields the trioxalatoferrate (III)

Reaction :- $Fe_2(SO_4)_3 + 3BaC_2O_4 \rightarrow Fe_2(C_2O_4)_3 + 3BaSO_4\downarrow$

$Fe_2(C_2O_4)_3 + 3K_2C_2O_4 \rightarrow 6H_2O \rightarrow 2K_3[Fe(C_2O_4)_3] \cdot 3H_2O$

Apparatus :- china dish (10cm diameter) watch glass, burner, water bath, 500ml beaker, buchner funnel, filter paper, water section pump vaccum dissicator etc.

Requirements :- Ferric sulphate crystals = 12.5 gm

Oxarium oxalate = 25 gm

Potassium oxalate, monohydrate = 13.6 gm

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Procedure:- Placed 12.5 g of ferric sulphate crystals, 25g of barium oxalate and 13.6g of potassium oxalate monohydrate in a 500ml beaker.

2. Added 300 ml water and stirred to dissolve the crystals.
3. Digested the above mixture for 1hr on water bath.
4. The reaction mixture shows formation of white ppt. Filtrate the mixture on buchner funnel using water suction pump.
5. Washed the precipitate three-four times with water.
6. Collected the filtrate in a china dish and evaporate it to half the volume by slow heating.
7. Allowed the solution to cool slowly keeping it undisturbed.
8. Light green crystals of trioxalatoferrate separate out. Decant off the supernatant liquid and transferred the crystals on filters.
9. Dried the crystals and recorded the yield.
10. Weighed the crystals and recorded the yield.

Calculation of Percentage Yields:-

The percentage yield can be calculated as follows:-

$Fe_2(SO_4)_3 + 3BaC_2O_4 + 3K_2C_2O_4 + 6H_2O \rightarrow 2K_3[Fe(C_2O_4)_3] + 3BaSO_4$

$[56\times2 + 3\times96]g = 400g$ $\qquad 2[39\times3 + 56 + 3\times64 + 3\times12] = 806\,g$

400 g of $Fe_2(SO_4)_3$ gives $K_3[Fe(C_2O_4)_3]\cdot 3H_2O = 806g$

12.5 g of $Fe_2(SO_4)_3$ will give $K_3[Fe(C_2O_4)_3]\cdot 3H_2O$

$$= \frac{806}{400} \times 12.5$$

$$= 25.2\,g$$

Theoritical yield = 25.2 g

Experimental yield = 12.6 g

Percentage yield $= \frac{12.6}{25.2} \times 100 = 200\,\%$

Precautions:- Do not reduce the volume of filtrate more than half of its original

2. Do not heat the filtrate strongly otherwise the crystals are not obtained in proper shape

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Experiment No. 3

Aim:- To prepare M/20 solution of ferrous ammonium sulphate (Mohr's salt). Using this solution find out the molarity and strength of the given $KMnO_4$ solution

Chemical Equations:-

Molecular equation

$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$

$[2FeSO_4(NH_4)_2SO_4 \cdot 6H_2O + H_2SO_4 + [O] \rightarrow Fe_2(SO_4)_3 + 2(NH_4)_2SO_4 + 13H_2O] \times 5$

$2KMnO_4 + 8H_2SO_4 + 10FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O \rightarrow K_2SO_4 + MnSO_4 + 5Fe_2(SO_4)_3 + 10(NH_4)_2SO_4 + 68H_2O$

Ionic equation

$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$

$[Fe^{2+} \rightarrow Fe^{3+} + e^-] \times 5$

$MnO_4^- + 8H^+ + 5Fe^{2+} \rightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$

Apparatus:- Pipette, titration flask, burette and test tubes

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Chemical Required :- Mohr's salt, $KMnO_4$, water, dilute sulphuric acid, indicator

Indicator :- $KMnO_4$ is self indicator

End Point :- Colourless to permanent pink colour ($KMnO_4$ in burette)

Procedure :- At first, we prepare 250ml of m/20 Mohr's salt solution by dissolving 4.9g of Mohr's salt in water. Then we rise the pipette with the m/20 Mohr's salt solution and pipette out 25 ml of it in a washed titration flask.

2. we rinse the burette and fill it with the given $KMnO_4$ solution

3. we add one the solution one test tube (in 20ml) full of dil. HCl (~4N) to the solution in the flask

4. Then we note the inital reading of the burette

5. Now we add $KMnO_4$ solution from the burette till a permanent light pink colour is imparted to the solution on addition of last single drop of $KMnO_4$. We note the final reading of the burette.

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Observations:-

Weight of Mohr's salt = 4.90 g
Volume of Mohr's salt prepared = 250 ml
Molarity of Mohr's salt solution = M/20
Volume of Mohr's salt taken for each titration = 25 ml

S.No	Volume of Mohr's Salt	Burette Reading			Average
		Initial	Final	Diff.	
1	25	0.7	25.9	25.2	
2	25	0.6	25.8	25.2	
3	25	0.3	25.3	25	25
4	25	0.5	25.5	25	
5	25	0.2	25.2	25	

Concordant Volume = 25 ml

Calculations:-

a) Molarity of $KMnO_4$ solution

From the balanced chemical equation, it is clear that 2 moles of $KMnO_4$ reacts with 10 moles of Mohr's salt.

$$\frac{M_{KMnO_4} \times V_{KMnO_4}}{M_{\text{Mohr's salt}} \times V_{\text{Mohr's salt}}} = \frac{2}{10} = \frac{1}{5}$$

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$$\frac{M_{KMnO_4} \times 25}{\frac{1}{20} \times 25} = \frac{2}{10}$$

$$\Rightarrow M_{KMnO_4} \times 20 = \frac{2}{10}$$

$$\Rightarrow M_{KMnO_4} = \frac{1}{100} = 0.01\,M$$

(6) Strength of $KMnO_4$ solution

Strength (in g/L) = Molarity × Molecular mass

$= 0.01 \times 158$

$= 1.58$ g/L

Experiment No. 4

Aim :- To prepare M/50 solution of oxalic acid with its help, determine the molarity and strength of the given solution of potassium permanganate ($KMnO_4$)

Chemical Equation :-

Molecular equation

$$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

$$COOH \cdot 2H_2O + [O] \xrightarrow{60\text{-}70^\circ C} 2CO_2 + 3H_2O \quad] \times 5$$

$$COOH$$

$$2KMnO_4 + 3H_2SO_4 + 5 \begin{matrix} COOH \\ | \\ COOH \end{matrix} \cdot 2H_2O \rightarrow K_2SO_4 + 2MnSO_4 + 18H_2O + 10CO_2$$

Ionic equation

$$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O \quad] \times 2$$

$$C_2O_4^{2-} \rightarrow 2CO_2 + 2e^- \quad] \times 5$$

$$2MnO_4^- + 16H^+ + 5C_2O_4^{2-} \rightarrow 2Mn^{2+} + 8H_2O + 10CO_2$$

Apparatus :- Burette, pipette, titration flask, 500ml measuring flask, test tube

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Chemicals Required :- oxalic acid crystals, $KMnO_4$, solution (1.26 gl/l), 8 N H_2SO_4

Indicator :- $KMnO_4$ is self indicator

END Point :- Colourless to Permanent pink colour ($KMnO_4$ in burette)

Procedure :- weighed exactly 1.26g of oxalic acid crystals and dissolved in water to prepare 500ml of its solution using in 500ml measuring flask - Rinsed the pipette with the oxalic acid solution and pipette out 20ml of it in a washed titration flask.

2. Rinsed and filled the burette with the given M/50 $KMnO_4$ solution
3. Added one test tube (20ml) full of dil H_2SO_4 (4N) to the solution in titration flask
4. Noted the final reading of the burette
5. Repeated the above step to get other concordant reading.

Observation :- weight of watched glass = 20 g
weight of (watch glass + oxalic acid) = 21.
weight of oxalic acid = 1.260g

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volume of oxalic acid solution prepared = 500 ml
solution taken in burette = M/50 $KMnO_4$
volume of oxalic acid taken for each titration = 20.0 ml

S.No	Initial Reading of Burette	Final Reading of burette	volume of $KMnO_4$ solution used
1	0 ml	13.9 ml	25 ml
2	14 ml	27 ml	25 ml
3	3 ml	18 ml	25 ml

Concordant volume = 25.0 ml

Calculations :-

a) Molarity of the $KMnO_4$ solution

From the overall balanced chemical equation it is clear that 2 moles of $KMnO_4$ reacts with 5 moles of oxalic acid

$$\frac{M_{KMnO_4} \times V_{KMnO_4}}{M_{oxalic\ acid} \times V_{oxalic\ acid}} = \frac{2}{5}$$

$$\frac{M_{KMnO_4}}{1/50} = \frac{2}{5} \times \frac{20}{1} \Rightarrow M_{KMnO_4} = \frac{2}{5} \times \frac{2}{5 \times 10} = \frac{2}{125} M$$

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b) Strength of the $KMnO_4$ solution = Molarity × Molecular mass

$= \frac{2}{25 \times 10} \times 158 = 2.528$ g/L

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Experiment No. 5

Aim :- To identify the basic and acid radicals in the given salt

Physical characteristics :-

state = Amarphous
colour = White
solubility = in dilute HCl

A) Dry Test For Basic Radi

Experiment observation Inferance :-

1) Dry Heating test
A pinch of salt is heated in a dry test tube
No gas evolved - NH_4^+ absent
No sublimate formed. May be absent
No change in colour
Zn^{2+}

2) Charcoal Cavity Test :-
A pinch of salt is mixed with double white residue and heated quantity of Na_2CO_3
No-colour change - Ca^{2+} Ba^{2+} Sr^{2+} may be present
which glows
Zn^{2+} absent

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and heated on a charcoal cavity in reading flame

Experiment	observation	Inference
3) Flame Test A paste of salt is prepared with conc. HCl and heated on flame	Brick red colour which doesn't persist	Ca^{2+} Present.

B Dry Test For Acid Ratical

Experiment	observation	Inferance
1) Dry Heating Test a) A pinch of salt is heated in a dry test tube with dil H_2SO_4	colourless, odourless gas which turns lime water milky	CO_3^{2+} or SO_3^{2-} may be present.
b) The gas evolved in the previous test is passed through acidified $K_2Cr_2O_7$ solution	solution doesn't turn green	SO_3^{2-} absent

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2) Again dry heating test is conducted but with conc H_2SO_4	No effect	Cl^- Br^- I^- absent CO_3^{2-} Present

Wet Test For Basic Radical

Experiment	observation	Inference
1) The given salt is soluble in dil HCl		Group -I absent
2) H_2S gas is passed through the solution obtained from Exp-I	no ppt	Group - II absent
3) Solution obtained from Exp - II was boiled to remove H_2S completely and finally a solution of NH_4Cl and NH_4OH is added	no ppt	Group - III absent
4) H_2S gas is passed through the solution obtained from Exp-III	no ppt	Group - IV absent

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5) The solution obtained from Exp-IV is heated to remove H_2S gas completely and finally a solution of NH_4OH and $(NH_4)_2CO_3$ is added — white ppt. is obtained — Group V Present

Analysis of Group V

Experiment :- The ppt was dissolved in hot dilute CH_3COOH solution and divided into three parts.

Experiment	observation	Inference
a) Few drops of $K_2Cr_2O_4$ solution are added	no yellow ppt formed	Ba^{2+} absent
b) One mole of $(NH_4)_2SO_4$ solution is added and warmed	no white ppt formed	Sr^{2+} absent
c) 2 ml of Ammonium oxalate solution was added to the 3rd	a white ppt formed	Ca^{2+} presen

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position followed by a solution and then the wall of test-tube is scratched

d) flame test is conducted with the ppt. — Brick red colour which doesn't persist — Ca^{2+} present confirmed

Wet Test for Acid Radical

Experiment	Observation	Inference
1 5ml of salt solution acidified with dil HNO_3 is taken in test tube and then we add $AgNO_3$ solution	no ppt	Cl^-, Br^-, I^- absent
2) 5ml of salt solution acidified with dilute HCl is taken and then we add $BaCl_2$ solution	no ppt	SO_4^{2-} absent.

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3) The salt solution is taken in a test tube and then dilute HCl is added	colourless, odourless gas turns lime water milky	CO_3^{2-} may be present
	doesn't turn acidified $K_2Cr_2O_7$ solution green	SO_3^{2-} absent.
4) The salt solution is acidified with acetic acid and then lead acetate solution is added	no ppt	S^{2-} absent.
5) The salt solution is acidified with dilute HCl and then $MgSO_4$ solution is added	white ppt obtained	CO_3^{2-} present.

Result :- The dry and wet tests conducted for the identification of the acid and basic radical conform that the salt contains Ca^{2+} as the basic radical and CO_3^{2-} as the acid radical

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Experiment No. 6

Aim:- To identify the Basic and acid radicals Present in the given salt

Physical characteristics:-

State - crystalline
Colour - White
Solubility - in cold water

A) Dry Test for Basic Radicals

Experiment	observation	Inference
1) Dry Heating test A pinch of salt is heated in a dry test tube	No gas evolved No Sublimate formed	NH_4^+ absent
	change of colour	Zn^{2+} may be present.

2) Charcoal cavity Test

A pinch of salt is mixed with doubled

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the quantity of Na_2CO_3 acid heated on a charcoal cavity in reducing flame	In hot condition colour change to yellow. In cold condition colour change to white	Zn^{2+} may be Present.

3) Flame Test

A pasle of salt prepared with conc HCl and heated on Flame	Green flashes	Zn^{2+} Present.

B) Dry Test For Acid Radicals

Experiment	observation	Interance
1) Dry Heating test A pinch of salt is heated in a dry test tube with dilute H_2SO_4	No effect	CO_3^{2-}, SO_3^{2-}, S^{2-} NO_2^- absent
2) Again dry heating test is conducted but with conc. H_2SO_4	No effect	Cl^-, Br^-, I^- absent

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3) A small amount of salt is boiled with dilute HCl in a test tube we filter the contents and to the filtrate add few drops of $BaCl_2$ solution	White ppt insoluble in conc. HCl	SO_4^{2-} present

Wet Test for Basic Radical

Experiment	observation	Inferance
1) The given salt is soluble in dil HCl		Group I absent.
2) H_2S gas is passed through the solution obtained from Exp 1.	no ppt	Group II absent
3) Solution obtained from Exp. 2 was boiled to remove H_2S completely and finally a solution of NH_4Cl and NH_4OH is added	no ppt	Group III absent.

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4) H_2S gas is passed through the solution obtained from Exp-3 — Dull white ppt is obtained — Group IV present (Zn^{2+})

Confirmation of Zn^{2+}

Experiment	Observation	Inference
1) Sodium Hydroxide Test To one part of original solution we add sodium hydroxide solution dropwise	white ppt. formed which dissolve on more addition of NaOH	
2) Pot. Ferrocyanide Test To another part we add pot ferrocyanide test	white or bluish white ppt - Formed	Zn^{2+} confirmed
3) Charcoal Cavity Test charcoal cavity test is conducted with the salt	whit Greenish formed	

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D) Wet Test For Acid Radical :-

Experiment	Observation	Inference
1) 5ml of salt solution acidified with dilute HNO_3 is taken in a test tube and then we adde $AgNO_3$ solution	no ppt	Cl^-, Br^-, I^- absent
2) The salt solution is taken in a test tube and then dil. HCl is added	no gas evolved	CO_3^{2-}, SO_3^{2-} absent.
3) The salt solution is acidified with acetic acid and then lead acetate sol. is added	no ppt	S^{2-} absent.

Result :- The Dry and wet tests conducted for the identification of the acidic and basic radicals confirm that the salt contains Zn^{2+} and SO_4^{2-} as the basic and acid radicals respectively.

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Experiment No. 7

Aim :- To identify the acid and basic radicals present in the given salt

Physical Characteristics :-

state - Crystalline
colour - white
solubility - water

A Dry Test For Basic Radical

Experiment	observation	Inference
1) Dry Heating Test A pinch of salt is heated in a dry test tube	A gas evolved colourless with characteristics small gives white fumes white sublimate formed	NH_4^+ may be present
2) A pinch of salt is mixed with double the quantity of Na_2CO_3 and heated on a charcoal cavity in reducing flame	no action	Zn^{2+}, Pb^{2+} Cu^{2+}, Ba^{2+} absent.

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B) Dry Test For Acid Radical

Experiment	observation	Inference
1) Dry Heading Test A pinch of salt is heated in a dry test tube with dilute HCl	no effect	CO_3^{2-}, CO_3^{2-}, C^{2-} absent.
2) Again dry heating test is conducted, but with con. H_2SO_4	Colourless gas with pungent small, white fumes with eg. Ammonia, white ppt. with $AgNO_3$	Cl^- present

C) Wet Test For Basic Radical

1) The solid salt is heated with conc. solution of NaOH	characteristic ammonia small gas gives white fumes when rod dipped in dil. HCl is brought near it	Group zero present.

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2) When passed through Nessler's — gives brown ppt — Group zero present (NH_4^+) confirmed.

D) Wet Test For Acid Radical

1) 5 ml of salt solution acidified with dil HNO_3 is taken in a test tube and then we add $AgNO_3$ solution — white ppt soluble in NH_4OH — Cl^-, Br^-, I^- present.

2) We heat a pinch of salt with small quantity of MnO_2 and conc. H_2SO_4 — Greenish yellow gas pungent irritating smell turns starch iodide paper blue — Cl^- may be present

Result :- Therefore the tests shows that the salt contains NH_4^+ and Cl^- as basic and acid radicals respectively.

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Experiment No. - 8

Aim :- To identify the functional group of a given organic compound.

Experiment	Observation	Inferance
1 Test For Unsaturation dissolved 0.2 ml of orga. nic compound in 2ml ccl4. Then added bromine water dropwise	Brown colour of bromine not discharged	No unsaturation is present
2) Test For Carboxylic Group:- Added a pinch of $NaHCO_3$ to 0.2ml of organic compound in a test tube	No effervescence	carboxylic group is absent.
3. Test For Phenolic Group. Added 0.2 ml of organic compound to 2-3 ml neutral $FeCl_3$ solution in a test tube	No green or violet colour obtained	Phenolic group obser

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4) Test For Alcoholic Groups

a) In a dry test tube 1ml of the given liquid is taken add about 1g of anhydrous calcium Sulphate and shake well to remove water Filter or decant off the liquid to another clean dry test tube and add a small piece of Na metal — A brisk effervescence is evolved — Alcohol is present.

5) Test For Carbonyl groups

Shake 0.2ml of organic compound with 2.3ml of 2,3-dinitrophenyl hydrazine — ~~Formed~~ No ppt is formed — Carbonyl group is absent.

Result :- The given organic compound contains alcoholic functional groups

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Experiment No. 9

Aim:- To identify the functional groups present in the given organic compound.

Experiment	observation	Inference
1) Test For unsaturation Dissolved 0.2 ml of organic compound in 2ml CCl_4. Then added Bc-water dropwise	Brown colour of bromine not discharged	No unsaturation is present
2) Test For Carboxylic Group Added a pinch of $NaHCO_3$ to 0.2 ml of organic compound in a test tube	No effervescence	Carboxylic group is present.
3) Test For Phenolic Group Added 0.2 ml of organic compound to 2-3 ml neutral $FeCl_3$ Solution test tube	No green or violet colour obtained	Phenolic group absent

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4. Test For Alcoholic group

In a dry test tube 1ml of the given liquid is taken add about 1g of anhydrous calcium sulphate. and shake well to remove water filled or decant off the liquid to another clean dry test tube and add a small piece of Na metal.

A brisk effervescence is evolved

Alcohole is present.

5) Test For Carbonyl Group

Shake 0.2ml of organic compound with 2.3ml of 2,3-dinitrophenyl hydrazine

No ppt is formed

Carbonyl group is absent.

Result:- The given organic compound contains aldehydic functional groups.

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Experiment No. 10

Aim :- To identify the functional groups present in the given organic compound

Experiment	Observation	Inference
1 Test For Unsaturation Dissolved 0.2 ml of organic compound in 2 ml CCl_4. then added bromine water dropwise	Brown colour of bromine not discharged	No unsaturation in present.
2) Test For Carboxylic Groups Carboxylic acid reacts with Sodium carbonate to given carbon dioxide gas which is to identify	A brisk effervence is produced	Presence of carboxylic groups
3) Test For Phenolic Group Added 0.2 ml of organic compound to 2-3 ml	No green or violet colour	Phenolic groups absent.

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neutral $FeCl_3$ solution obtained in a test tube

4. Test For Alcoholic Groups

Adde a small piace of Na to 1 ml of the given liquid in a dry test tube — No effervesence — Alcoholic group is absent.

5. Test For Carbonyl groups

A Shook 0.2 ml of organic compound with 2-3 ml of 2,3-dinitrophenyl hydrazine in a test tube — No ppt is formed — Carbonyl group is absent.

Result:- The given organic compound contains Carboxylic functional groups.

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