

I N D E X

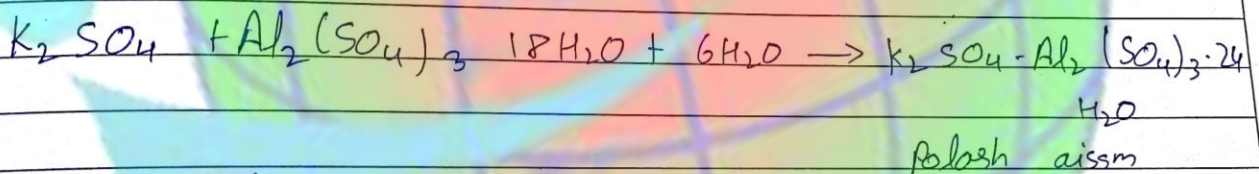
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Experiment No. 1

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

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 crystallization when octahedral crystals of potash alum separate out.



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

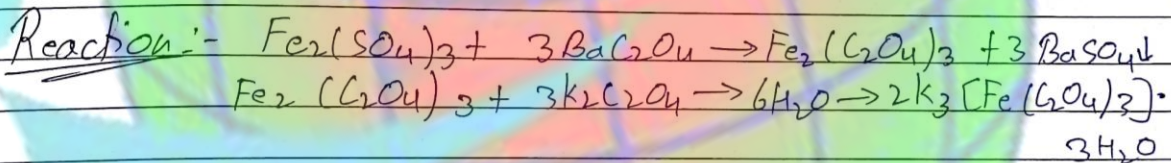
- 2) We will take other 250ml beaker wash it with water and then transferred 10g aluminium sulphate crystals to it. Added about 20ml of water and 1ml of dil H_2SO_4 to prevent hydrolysis of aluminium sulphate. Heated for about 5 min.
3. Mixed the two solution in a china-dish and placed the china dish on a wire gauge placed over a beaker. Stirred the solution with a glass rod. Concentrated the solution till crystallisation point is reached. Placed the dish over a beaker containing cold water.
4. Brown 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

Experiment No. 2

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

Theory :- The compound can be prepared by reacting 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 ferrisulphate (III)



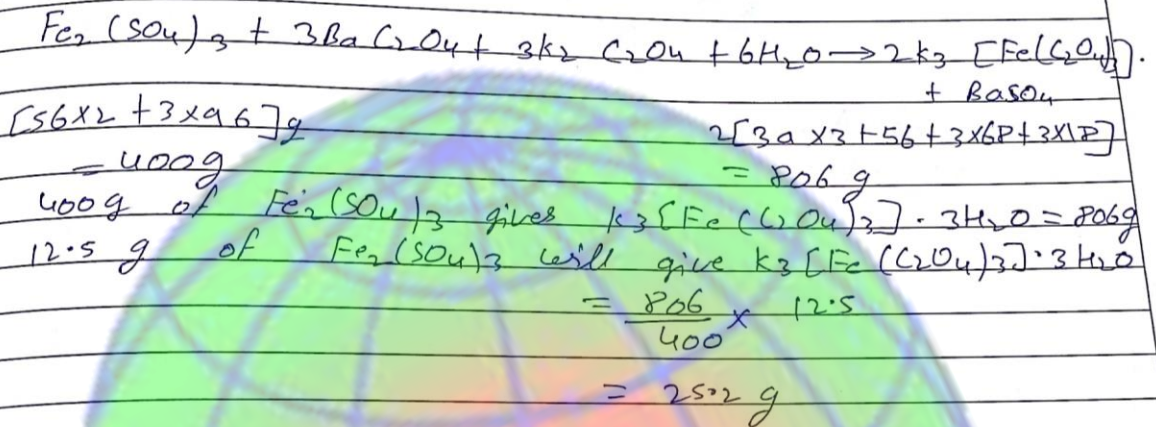
Apparatus :- china dish (10cm diameter) watch glass, beaker, water bath, 500ml beaker, buchner funnel, filter paper, water suction pump vacuum desiccation etc.

Requirements :- Ferric sulphate crystals = 12.5 gm
 Oxalic acid = 25 gm
 Potassium oxalate, monohydrate = 13.6 gm

- Procedure:- Weighed 12.5 g of ferric sulphate crystals, 25g of barium oxalate and 13.6g of potassium oxalate monohydrate in a 500ml beaker.
- Added 300 ml water and stirred to dissolve the crystals.
 - Diggled the above mixture for 1hr on water bath.
 - The reaction mixture shows formation of white ppt. filtered the mixture on buchner funnel using water suction pump.
 - washed the precipitate three-four times with water.
 - Collected the filtrate in a china dish and evaporated it to half the volume by slow heating.
 - Allowed the solution to cool slowly keeping it undisturbed.
 - Light green crystals of thionalsoterrate separate out. Decant off the supernatant liquid and transferred the crystals on filter.
 - Dried the crystals and recorded the yield.
 - weighed the crystals and recorded the yield.

Calculation of Percentage yields:-

The percentage yield can be calculated as follows:-



Theoretical yield = 25.2 g

Experimental yield = 12.6 g

Percentage yield = $\frac{12.6}{25.2} \times 100 = 50\%$

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

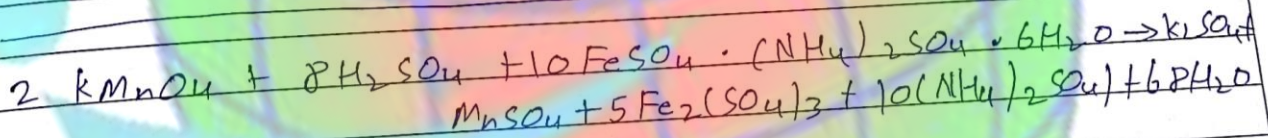
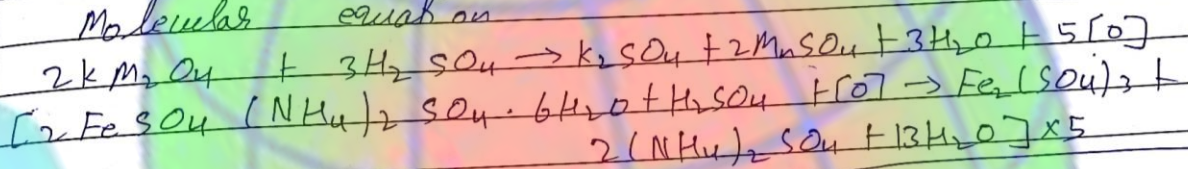
2. Do not heat the filtrate strongly otherwise the crystals are not obtained in proper shape

Experiment No. 3

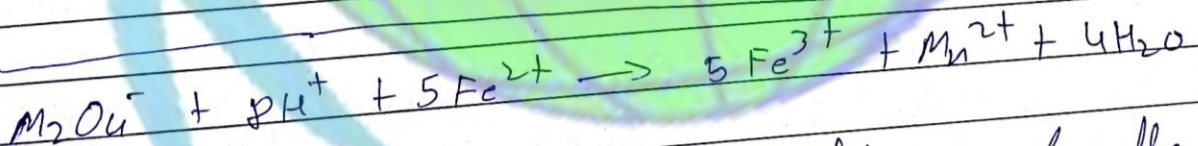
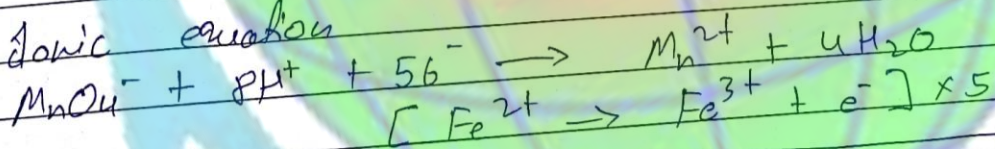
Aim:- To prepare m/20 solution of ferrous ammonium sulphate (Mohr's salt). Using this solution find out the molarity and strength of the given KMnO_4 solution.

Chemical Equations:-

Molecular equation



Ionic equation



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

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 250 ml of m/po Mohr's salt solution by dissolving 4.9 g of Mohr's salt in water. Then we rinse the pipette with the m/po 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 ferrous solution one test tube (in 20 ml) full of dil. HCl (2N) to the solution in the flask

4. Then we note the initial 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	25
2	25	0.6	25.8	25.2	
3	25	0.3	25.3	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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$$\frac{M_{\text{KMnO}_4} \times 25}{\frac{1}{20} \times 25} = \frac{2}{10}$$

$$\Rightarrow M_{\text{KMnO}_4} \times 20 = \frac{2}{10}$$

$$\Rightarrow M_{\text{KMnO}_4} = \frac{1}{100} = 0.01 \text{ M}$$

(6) strength of KMnO_4 solution

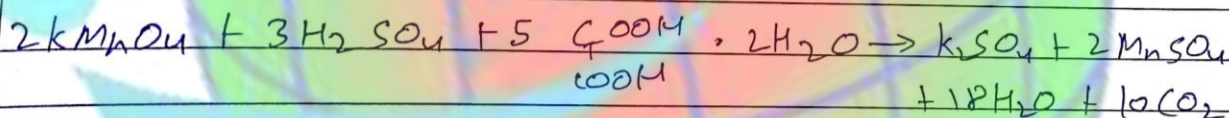
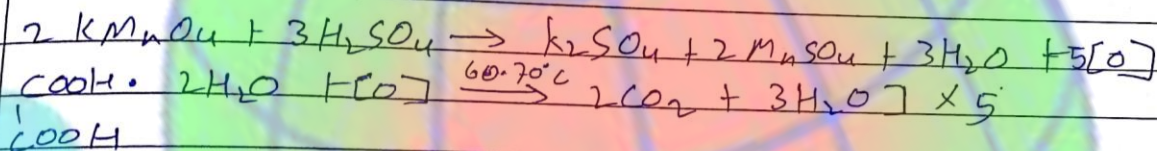
$$\begin{aligned} \text{Strength (in g/L)} &= \text{Molarity} \times \text{molecular mass} \\ &= 0.01 \times 158 \\ &= 1.58 \text{ g/L} \end{aligned}$$

Experiment No. 4

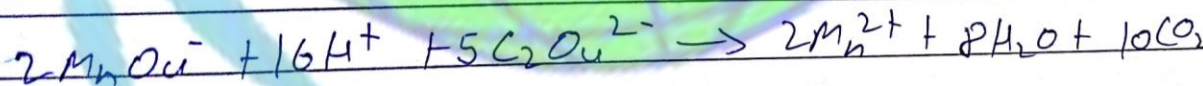
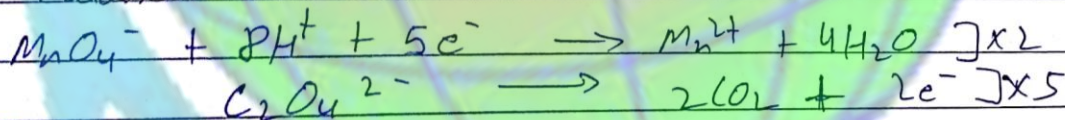
Aim :- To prepare m/50 solution of oxalic acid with its help, determine the molarity and strength of the given solution of potassium permanganate (KMnO_4)

Chemical Equation :-

Molecular equation



Ionic equation



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

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

Indicator :- KMnO_4 is self indicator

END Point :- colourless to permanent pink colour (KMnO_4 in burette)

Procedure :- weighed exactly 12.6g of oxalic acid crystals and dissolved in water to prepare 500ml of its solution using in 500ml measuring flask - Rinsed the pipette with this oxalic acid solution and pipette out 20ml of it in a washed titration flask.

2. Rinsed and filled the burette with the given $\text{M}/50$ 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 watch glass = 20 g
weight of (watch glass + oxalic acid) = 21.26 g
weight of oxalic acid = 1.26 g

Volume of oxalic acid solution prepared = 500 ml
 solution taken in burette = $\frac{1}{5}$ 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

$$\frac{M_{\text{KMnO}_4} \times V_{\text{KMnO}_4}}{M_{\text{oxalic acid}} \times V_{\text{oxalic acid}}} = \frac{2}{5}$$

$$\frac{M_{\text{KMnO}_4}}{\frac{1}{50} \times 20} = \frac{2}{5} \Rightarrow M_{\text{KMnO}_4} = \frac{2 \times 2}{5 \times 10} = \frac{2}{125} \text{ M}$$

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b) strength of the KMnO_4 solution = Molarity \times
molecular mass

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

Experiment No. 5

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

Physical characteristics :-

state = Amorphous

colour = white

solubility = in dilute HCl

A) dry Test For Basic Radi

Experiment observation Inference :-

1) dry Heating test

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

2) Charcoal cavity :-

Test

A pinch of salt is No-colour change - Ca^{2+} Ba^{2+} Sr^{2+}
 Mixed with double white residue may be present
 and heated quantity of Na_2CO_3 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	Bright red colour which doesn't persist	Ca^{2+} Present

B Dry Test For Acid Radical

Experiment	Observation	Inference
1) Dry Heating Test a) A pinch of salt is heated in a dry test tube with dil H_2SO_4	colourless, colourless 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 $\text{K}_2\text{Cr}_2\text{O}_7$ solution	solution doesn't turn green	SO_3^{2-} absent

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

- 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.

<u>Experiment</u>	<u>Observation</u>	<u>Inference</u>
a) Few drops of K_2CO_3 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) 2ml of Ammonium oxalate solution was added to the 3rd	a white ppt formed	Ca^{2+} present

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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 a
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.

3) The salt solution is taken in a test-tube and then dilute HCl is added. Colourless / colourless gas turns lime water milky. CO_3^{2-} may be present. Doesn't turn acidified $\text{K}_2\text{Cr}_2\text{O}_7$ 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 test that the salt contains Ca^{2+} as the basic radical and CO_3^{2-} as the acid radical.

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

1) Dry Test for Basic Radicals

Experiment	Observation	Inference
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1) Dry Heating test

A pinch of salt is heated in a dry test tube	No gas evolved No sublimate formed	NH_4^+ absent
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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 part of salt prepared with conc. HCl and heated on flame

Green flashes

Zn^{2+} present.

B) Dry test For Acid Radicals

Experiment

observation

Inference

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-}
None absent

2) Again dry heating test is conducted but with conc. H_2SO_4

No effect

Cl^- , Br^- , I^-
absent

- 3) A small amount of salt is boiled with dilute HCl in a test tube and the contents are filtered. 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

<u>Experiment</u>	<u>observation</u>	<u>Inference</u>
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1) The given salt is soluble in dil HCl		Group I absent.
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2) H_2S gas is passed through the solution obtained from Exp 1.	no ppt	Group II absent
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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. Ferricyanide Test
To another part we add pot ferricyanide test

white or bluish white ppt. formed

Zn^{2+} confirmed

3) Charcoal cavity Test
Charcoal cavity test is conducted with the salt

white greenish formed

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Q) 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 add 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.

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 Day Test For Basic Radical

Experiment

Observation

Inference

1) Day Heating Test

A pinch of salt is heated in a dry test tube

A gas evolved colourless with characteristics smell 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 Heating 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 conc. H_2SO_4

colourless gas with pungent smell, white fumes with e.g. 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 smell gas gives white fumes when rod dipped in dil. HCl is brought near it

Group zero present.

2) When passed through Nessler's gives brown ppt Group 2 also 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 cation and acid radicals respectively.

Experiment No.-8

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

<u>Experiment</u>	<u>Observation</u>	<u>Inference</u>
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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 is present
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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 absent
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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 absent
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4) Test For Alcohols Group

4) 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 group

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

Experiment No. 9

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

<u>Experiment</u>	<u>observation</u>	<u>Inference</u>
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1) Test For Unsaturation

Dissolved 0.2 ml of organic compound in 2 ml 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 in 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. 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 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.

Experiment No. 10

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

<u>Experiment</u>	<u>Observation</u>	<u>Inference</u>
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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 is present.
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2) Test For Carboxylic Group

Carboxylic acid reacts with sodium carbonate to give carbon dioxide gas which is to identify	A brisk efferece is produced	Presence of carboxylic group
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3) Test For Phenolic Group

Added 0.2 ml of organic compound to 2-3 ml	No green or violet colour	Phenolic group absent.
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neutral FeCl₃ solution
in a test tube obtained

4. Test For Alcoholic Groups

Add a small piece
of Na to 1 ml of the
given liquid in a dry
test tube

No effervescence

Alcoholic
group is
absent.

5. Test For Carbonyl group

Shook 0.2 ml of organic
compound with 2-3 ml
of 2,3-dimethylphenyl
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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