ENGINEERING CHEMISTRY LABORATORY MANUAL FOR DIPLOMA STUDENTS

Prepared By :-

Kishan Kumar Agrawal
M.Sc., CSIR NET- JRF
Lect. In Chemistry
Govt. Polytechnic, Balangir

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PREPARATION AND STUDY OF PHYSICAL AND CHEMICAL PROPERTIES OF CO_2 GAS

Aim of the experiment :

To prepare carbon dioxide in the laboratory and study it's physical and chemical properties .

Apparatus and Chemicals Required :

Woulf's bottle
Gas jar with lid
Marble chips
Dilute hydrochloric acid

Procedure :

1. Put some marble chips in a Woulf's bottle and fit the bottle with a thistle funnel and a delivery tube.

2. Place a collecting jar at the other end of the delivery tube.

3. Pour dilute hydrochloric acid in the bottle through the thistle funnel so that the marble chips are covered with the acid.

4. Collect the gas jars and cover them.

Observation :

Effervescence takes place in the bottle and the gas is collected by upward displacement of air.

The properties of the gas formed are as follows.

1. It is colourless.

2. It has a faint odour.

3. It dissolves in water immediately.

4. It is heavier than air.

5. If you put a lighted splinter in a jar containing carbon dioxide gas, it extinguishes. The gas does not support combustion.

6. If you put a wet blue litmus paper into the jar, it turns red. The gas is therefore acidic.

7. If add some limewater to the jar and shake, it turns milky.

8. If you put a burning magnesium ribbon in the jar, it burns with a dazzling light. This is because an oxide of magnesium is formed by reacting with carbon dioxide.

Precautions :

1. Check that the thistle funnel dips into the acid.
2. Do not leave the gas jars uncovered

PREPARATION AND STUDY OF PHYSICAL AND CHEMICAL PROPERTIES OF NH_3 GAS

Aim of the experiment :

To prepare ammonia in the laboratory and study it's physical and chemical properties

Apparatus Required :

Hard glass test tube
Gas jar with lid
Bunsen burner
Delivery tube
Clamp stand
Cork

Chemicals Required :

Solid ammonium chloride (NH_4Cl)

Quick lime (CaO) or dry slaked lime $Ca(OH)_2$

Procedure :

1) A certain mass of NH4Cl & double of it's mass of Ca(OH)2 is taken in a boiling tube.
2) A delivery tube is fitted to the mouth of the boiling tube using a cork, & the boiling tube is clamped to a stand.
3) The open end of the delivery tube is fitted to the lower end of a tower containing dry CaO, which is being used here as a drying agent.
4) Finally, the side tube at the upper part of the tower is inserted into a dry & inverted gas jar.
5) Now, if the mixture in the boiling tube is heated slowly, wet NH3 is produced
6) Ammonia gas formed here is collected by downward displacement of air.
7) Finely divided iron or alumina can be used as catalyst in this reaction.

Observation :

The properties of the gas formed are as follows.

1. It is colourless.

2. It has a distinct odour.

3. It dissolves in water immediately.

4. It is heavier than air.

5. If you show a glass rod dipped in conc. HCl to the gas, it will produce white fumes due to the formation of NH_4Cl .

6. If you put a wet red litmus paper into the jar, it turns blue. The gas is therefore basic.

7. If you pass the gas through 2 cc of Nessler's reagent in a clean dry test tube it turns brown .

8. If you slowly pass the gas through 2cc of aqueous copper sulphate solution ina clean dry test tube, bluish white ppt is formed..

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CRYSTALLIZATION OF COPPER SULPHATE

Aim of the experiment :

To prepare crystals of copper sulphate from copper carbonate.

Apparatus and Chemicals Required :

China dish
Glass rod
Wash bottle
Copper carbonate
Burner
250 ml beaker
Wire gauze
Tripod stand
Distilled water
Conc. Sulphuric acid

Procedure :

1. Take a clean beaker (250 ml) and put the powdered sample of copper carbonate in it.

2. Add distilled water and stir the contents gently with the help of a glass rod.

3. In order to make the solution more clear add two or three drops of concentrated sulphuric acid in it.

4. Heat the solution in the beaker to 60-70^{0}C on a wire gauze.

5. Stir it continuously and add more impure copper sulphate until no more of it dissolves.

6. Filter the solution and collect the filtrate in a china dish.

7. Place the china dish over wire gauze kept over a tripod stand and heat it gently (do not boil).

8. As the solution gets heated, stir it with a glass rod. This helps in uniform evaporation and prevents the formation of a solid crust.

9. When the volume of the solution is reduced to one-half, take out a drop of the concentrated solution on one end of the glass rod and cool it by blowing air. Formation of thin crust indicates that crystallization point is reached.

10. Turn off the burner, cover the dish with a watch glass, and keep it undisturbed. As the solution cools down, crystals separate out. Slow cooling ensures better crystallization.

11. Decant the mother liquor and wash the crystals with a thin stream of cold water with the help of a wash bottle.

12. Dry the crystals by pressing them gently between sheets of filter paper.

Observation :

Blue colored crystals of copper sulphate ($CuSO_4.5H_2O$) are formed.

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Acid-Base Titration

Aim of the experiment :

To determine the strength of the given sodium hydroxide solution using N/10 hydrochloric acid solution.

Theory :

Here, the sodium hydroxide solution is taken in burette and a known volume (10.0 ml) of the N/10 hydrochloric acid solution is taken in the titration flask.The titration is carried out using phenolphthalein as indicator.

Apparatus and Chemicals Required :

Burette
Burette stand
Pipette
250 ml conical flask
Sodium hydroxide (unknown strength)
N/10 Hydrochoric acid
Phenolpthalein indicator

Procedure :

1. Take a burette and wash it with water.

2. Rinse and then fill the burette with the given sodium hydroxide solution. Clamp it vertically in burette stand.

3. Rinse the pipette with the given N/10 hydrochloric acid solution.

4. Pipette out 10 ml of the hydrochloric acid solution in a washed titration flask.

5. Add 1-2 drops of phenolphthalein indicator into it and place it just below the nozzle of the burette over a white glazed tile.

6. Note down the lower meniscus of the solution in the burette and record it as the initial burette reading.

7. Now run the sodium hydroxide solution slowly and dropwise into the flask till a very faint permanent pink colour is just obtained. Read the lower meniscus of the solution again in the burette and record it as final burette reading.

8. Repeat the procedure until three concordant readings are obtained.

Observations :

S.No.	Initial reading of the burette	Final reading of the burette	Volume of the sodium hydroxide solution used
1.	—	_	— ml
2.	—	—	— ml
3.	—	—	— ml
4.	—	—	— ml

Concordant volume = x ml (say)

Calculations :

Normality of the given hydrochloric acid solution = N/10 (N_1)
Volume of hydrochloric acid solution taken for each titration = 10 ml (V_1)

Normality of the given sodium hydroxide solution = let N_2
Volume of sodium hydroxide solution(obtained from titration) = x ml (V_2).

According to the equation,

$$N_1V_1 = N_2V_2$$

Result :

The strength of the given sodium hydroxide solution is found to be …….

QUALITATIVE ANALYSIS

Analytical chemistry deals with qualitative and quantitative analysis of the substances. In inorganic qualitative analysis, the given compound is analyzed for the radicals, i.e., cation and the anion, that it contains. The common procedure for testing any unknown sample is to make its solution and then test this solution for the ions present in it. There are separate procedures for detecting cations and anions, therefore qualitative analysis is studied under cation analysis and anion analysis. The systematic procedure for qualitative analysis of an inorganic salt involves the following steps :

(a) Preliminary tests
1. Physical appearance (colour and smell).
2. Dry heating test.
3. Charcoal cavity test.
4. Charcoal cavity and cobalt nitrate test.
5. Flame test.
6. Borax bead test.
7. Dilute acid test.
8. Potassium permanganate test.
9. Concentrated sulphuric acid test.
10. Tests for sulphate, phosphate and borate.

(b) Wet tests for acid radical.
(c) Wet tests (group analysis) for basic radical.

Physical Examination Of The Salt

The physical examination of the unknown salt involves the study of colour, smell and density. The test is not much reliable, but is certainly helpful in identifying some coloured cations.

Note :

1. If you have touched any salt, wash your hands at onte. It may be corrosive to skin.
2. Never taste any salt, it may be poisonous. Salts of arsenic and mercury are highly poisonous.
3. Salts like sodium sulphide, sodium nitrite, potassium nitrite, develop a yellow colour.

Physical Examination

Experiment	Observations	Inference
1. Colour	Blue or Bluish green Light green Dark brown Green Pink Light pink, flesh colour or earthy colour White	Cu^{2+} Fe^{2+} Fe^{3+} Ni^{2+} Co^{2+} Mn^{2+} Shows the absence of Cu^{2+}, Fe^{2+}, Fe^{3+}, Ni^{2+}, Mn^{2+}, CO^{2+}
2. Smell Take a pinch of the	Ammoniacal smell Vinegar like smell	NH_4^+ $CH3COO^-$

salt between your fingers and rub with a drop of water	Smell like that of rotten eggs	S^{2-}
3. Density	(i) Heavy (ii) Light fluffy powder	Salt of Pb^{2+}, or Ba^{2+} Carbonate
4. Deliquescence	Salt absorbs moisture and becomes paste like	(i) If coloured, may be $Cu(NO_3)_2$, $FeCl_3$. (ii) If colourless, may be $Zn(NO_3)_2$, chlorides of Zn^{2+}, Mg^{2+} etc.

Dry Heating Test

This test is performed by heating a small amount of salt in a dry test tube. Quite valuable information can be gathered by carefully performing and noting the observations here. On heating some salts undergo decomposition thus evolving the gases or may undergo characteris¬tic changes in the colour of residue. These observations are tabulated below along with the inferences that you can draw.

Dry Heating Test

Observations	Inference
1. Gas evolved	CO_3^{2-} or $C_2O_4^{2-}$
(a) **Colourless and odourless gas**	
CO_2 gas turns lime water milky	
(b**) Colourless gas with odour**	Hydrated S^{2-}
(i) H_2S gas—Smells like rotten eggs, turns	
lead acetate paper black.	SO_3^{2-}
(ii) SO_2 gas—Smells like burning sulphur,	
turns acidified potassium dichromate paper green	
(iii) HCl gas—Pungent smell, white fumes with	Cl^-
ammonia, white ppt with silver nitrate solution.	
(iv) Acetic acid vapours—Characteristic vin-	

egar like smell. (v) NH_3 gas—Characteristic smell, turns Nessler's solution brown. (c) **Coloured gases**—Pungent smell (i) NO_2 gas—Reddish brown, turns ferrous sulphate solution black. (ii) Cl_2 gas—Greenish yellow, turns starch io- dide paper blue. (iii) Br_2 vapours—Reddish brown, turns starch paper orange yellow. (iv) I_2 vapours—Dark violet, turns starch paper blue.	CH_3COO^- NH_4^+ NO^{2-} or NO^{3-} Cl^- Br^- I^-
2. Sublimate formed (a) White sublimate	NH_4^+ I^-

(b) Black sublimate accompanied by violet vapours	
3. Decrepitation The salt decrepitates.	A salt having no water of crystallisation. For example, $Pb(NO_3)_2$, NaCl, KBr.
4. Swelling The salt swells up into voluminous mass.	PO_4^{3-} indicated
5. Residue (i) Yellow when hot white when cold (ii) Brown when hot and yellow when cold (iii) White salt becomes black on heating (iv) White residue, glows on heating (v) Original salt blue becomes white on heating (vi) Coloured salt becomes brown or black on heating.	 Zn^{2+} Pb^{2+} CH_3COO^- indicated Ba^{2+}, Sr^{2+}, Ca^{2+}, Mg^{2+}, etc. Hydrated $CuSO_4$ indicated CO^{2+}, Cu^{2+}, Mn^{2+} indicated.

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Charcoal Cavity Test

This test is based on the fact that metallic carbonates when heated in a charcoal cavity decom¬pose to give corresponding oxides. The oxides appear as coloured incrustation or residue in the cavity. In certain cases, the oxides formed partially undergo reduction to the metallic state producing metallic beads or scales.

Examples :

(a) $ZnSO_4 + Na_2CO_3 \longrightarrow ZnCO_3 + Na_2SO_4$

$ZnCO_3 \longrightarrow ZnO + CO_2 \uparrow$

Yellow when hot, white when cold

(b) $Pb(NO_3)_2 + Na_2CO_3 \longrightarrow PbCO_3 + 2NaNO_3$

$PbCO_3 \longrightarrow PbO + CO_2 \uparrow$

$PbO + C \longrightarrow Pb + CO \uparrow$

Bead

(c) $CuSO_4 + Na_2CO_3 \longrightarrow CuCO_3 + Na_2SO_4$

$CuCO_3 \longrightarrow CuO + CO_2 \uparrow$

$CuO + C \longrightarrow Cu + CO \uparrow$

Reddish scales

Procedure

While performing charcoal cavity test, make a small cavity on a charcoal block with the help of borer. Mix small amount of salt with double its quantity of sodium carbonate. Place it in the cavity made on the block of charcoal. Moisten with a drop of water and direct the reducing flame of the bunsen burner on the cavity by means of a mouth blowpipe.Heat strongly for sometime and draw inference.

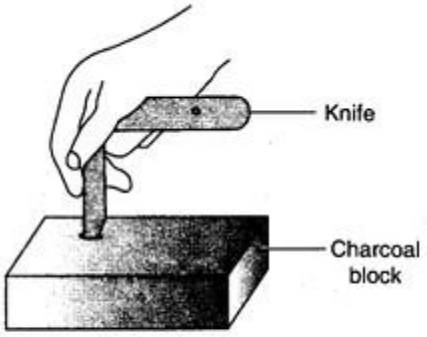


Fig. 9.2. Making bore on a charcoal block.

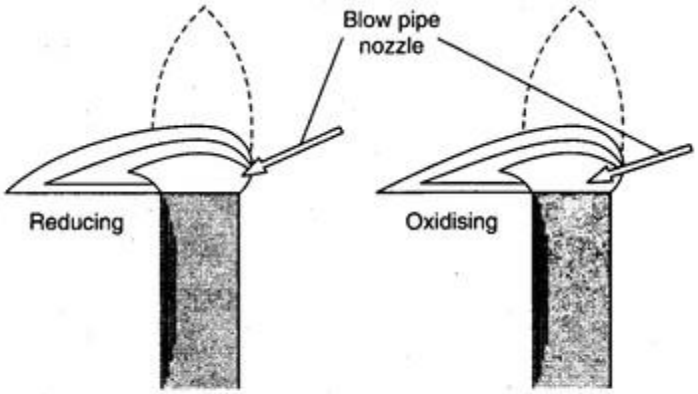


Fig. 9.3. Directing flame with blow pipe.

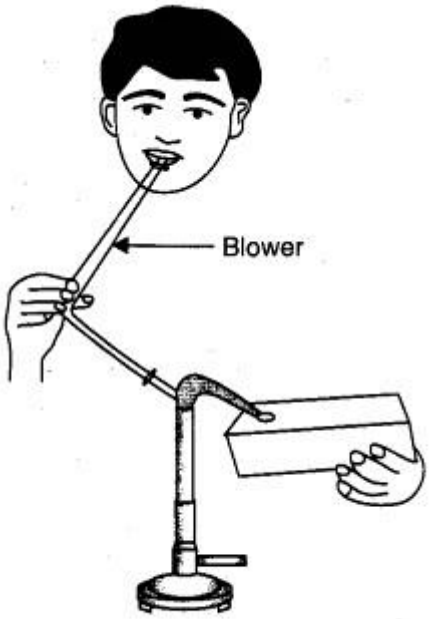


Fig. 9.4. Blowing flame on the cavity.

Charcoal Cavity Test

Observations			**Inference**
Incrustation or Residue		**Metallic bead**	
Hot	**Cold**		
Yellow	White	None	Zn^{2+}
Brown	Yellow	Grey bead which marks the paper	Pb^{2+}
None	None	Red beads or scales	Cu^{2+}
White residue which glows	None	None	Ba^{2+}, Ca^{2+}, Mg^{2+}
Black	None	None	Nothing definite—generally coloured s

To obtain a reducing flame with the help of a mouth blow pipe, make the bunsen burner flame luminous by closing the air holes of the burner. Keep the nozzle of the blow pipe just outside the flame and blow gently on to the cavity.

Cobalt Nitrate Test

This test is applied to those salts which leave white residue in charcoal cavity test. The test is based on the fact that cobalt nitrate decomposes on heating to give cobalt oxide, CoO. This combines with the metallic-oxides, present as white residue in the charcoal cavity forming coloured compounds. For example, when a magnesium salt undergoes charcoal cavity test, a white residue of MgO is left behind. This on treatment with cobalt nitrate and subsequent heating forms a double salt of the formula MgO.CoO which is pink in colour. In addition to metallic oxides, phosphates and borates also react with cobalt oxide to form $Co_3(PO_4)_2$ and $Co_3(BO_3)_2$ which are blue in colour. Some of the reactions involved are given below :

$$2Co(NO_3)_2 \xrightarrow{\Delta} 2\ CoO + 4NO_2 + O_2$$

(i) Zinc salt :

$$ZnO + CoO \longrightarrow ZnO.CoO\ (Green)$$

(ii) Magnesium salt :

$$MgO + CoO \longrightarrow MgO.CoO\ (Pink)$$

Procedure

Put one or two drops of cobalt nitrate solution on the white residue left after charcoal cavity test. Heat for one or two minutes by means of a blow pipe in oxidising flame. Observe the colour of the residue and draw inferences.

Cobalt Nitrate-Charcoal Cavity Test

Color of the Residue	**Inference**
Green	Zn^{2+}
Pink	Mg^{2+}

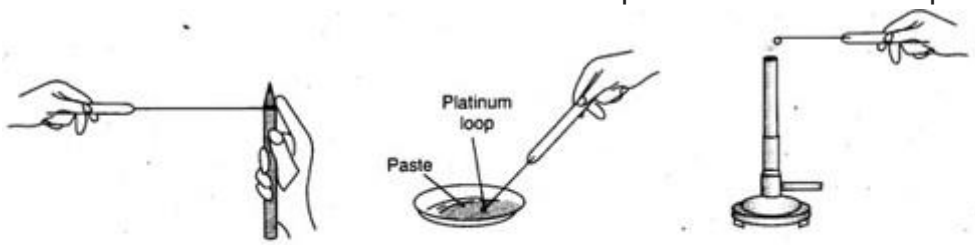
Blue	PO_4^{3-}
Black	It is due to the formation of CoO. No definite indication.

Note:

1. Perform this test only if the residue in the charcoal cavity test is white.
2. Do not put more than 2 drops of cobalt nitrate on the white residue. Excess cobalt nitrate may decompose to give cobalt oxide which is black in colour.
3. Use dilute solution of cobalt nitrate.

Flame Test

Certain salts on reacting with cone. HCl from their chlorides, that are volatile in non-luminous flame. Their vapours impart characteristic colour to the flame. This colour can give reliable information of the presence of certain basic radicals. For proceeding to this test, the paste of the mixture with cone. HCl is introduced into the flame with the help of platinum wire.



(a) Making a loop of platinum wire. (b) Dipping the platinum wire in the paste of salt and HCl. (c) Introducing the wire in the flame.

Fig. 9.5. Flame test.

Procedure

Clean the platinum wire by dipping it in some cone. HCl taken on a watch glass and then heating strongly in the flame. This process is repeated till the wire imparts no colour to the flame. Now prepare a paste of the salt with cone. HCl on a clean watch

glass. Place small amount of this paste on platinum wire loop and introduce it into the flame. Note the colour imparted to the flame with naked eye and through blue glass.

Flame Test

Color of the Flame		**Inference**
With naked eye	**Through blue glass**	
1. Brick-red (not persistent)	Light yellowish green	Ca^{2+}
2. Crimson-red (persistent)	Crimson	Sr^{2+}
3. Violet	Violet	K^{+}
4. golden yellow	white	Na^{+}
5. Green flashes		Zn^{2+} and Mn^{2+} salts
6. Dull bluish-white	White	Pb^{2+}

Test for Acid Radicals and Basic Radicals (Unknown)

Experiment	Observations	Inference
1. Physical examination:		
(a) Noted the colour of the given salt.	White	Cu^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}, Fe^{3+} absent.
(b) Noted the smell of the salt.	No specific odour	NH_4^+, S^{2-} and CH_3COO^- may be absent.
2. Dry heating test		
Heated a pinch of the salt in a dry test tube and noted the following observations :		
(a) Gas evolved	A reddish brown gas evolved which turned $FeSO_4$ solution black.	$N0^{3-}$ may be present.
(b) Sublimation	No sublimate formed.	Ammonium halides, aluminium chloride, iodide may be absent.

(c) Decrepitation (d) Colour of the residue	No crackling sound observed. White	Lead nitrate, bariumnitrate may be absent. Ba^{2+}, Sr^{2+}, Ca^{2+}, Al^{3+}, Mg^{2+}, etc., may be present.
3. Charcoal cavity test Mixed a pinch of the salt with double the quantity of Na_2CO_3 and heated the mixture on a charcoal cavity in the reducing flame.	White residue.	Ba^{2+}, Sr^{2+}, Ca^{2+}, Al^{3+}, Mg^{2+}, etc., may be present.
4. Cobalt nitrate test To the above white residue added a drop of cobalt nitrate solution. Heated it in oxidising flame.	No characteristic colour.	Al^{3+}, Zn^{2+},,Mg^{2+}, PO_4^{3-}, may absent.
5. Flame test Prepared a paste of the salt with cone. HCl and performed flame test.	Brick red colour appeared	Ca^{+2} may be present.
7. Dil. sulphuric acid test Treated a pinch of the salt with dil. H_2SO_4 and warmed.	No gas evolved.	CO_3^{2-}, S^{2-}, SO_3^{2-}, NO_2^- may absent.

8. $KMnO_4$ test To a pinch of the salt added dil. H_2SO_4 and a drop of $KMnO_4$ solution.	Pink colour of $KMnO_4$ was not discharged.	Cl^-, Br^-, I^-, $C_2O_4^{2-}$ and Fe^{2+} r be absent.
9. Cone, sulphuric acid test Heated a pinch of the salt with cone, sulphuric acid and added to it a paper pellet.	A reddish brown gas evolved which turned $FeSO_4$ solution black.	NO_3^- may be present.
10. Confirmatory test for ni-trate (a) Copper chips test. Heated a pinch of the salt with cone, sulphuric acid and a few copper chips. (b) Ring test. To 2-3 ml of the salt solution, added freshly pre-pared $FeSO_4$ solution. Then added cone, sulphuric acid along the sides of the test tube.	Reddish brown gas evolved. A dark brown ring formed at the junction of the two liquids.	NO_3^- confirmed. NO_3^- confirmed.

Result

Acid radical : NO_3^-
Basic radical : Ca^{2+}

Test for Unknown salt composed of one Acid Radical and one Basic Radical

Experiment	Observations	Inference
1. Physical examination: (a) Noted the colour of the given salt. (b) Noted the smell of the salt.	White No specific odour	Cu^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}, Fe^{3+} absent. NH_4^+, S^{2-} and CH_3COO^- may be absent.
2. Dry heating test Heated a pinch of the salt in a dry test tube and noted the following observations : (a) Gas evolved (b) Sublimation	A colorless gas evolved which turned lime water milky. No sublimate formed.	CO_3^{2-} may be present. Ammonium halides, aluminium chloride, iodid may be absent.

(c) Decrepitation (d) Colour of the residue	No crackling sound observed. Yellow when hot and white when cold	Lead nitrate, barium nitrate, may be absent. Zn^{2+} may be present.
3. Charcoal cavity test Mixed a pinch of the salt with double the quantity of Na_2CO_3 and heated the mixture on a charcoal cavity in the reducing flame.	Yellow when hot and white when cold	Zn^{2+} may be present.
4. Cobalt nitrate test To the above white residue added a drop of cobalt nitrate solution. Heated it in oxidising flame.	Green Residue	Zn^{2+} may be pesent.
5. Flame test Prepared a paste of the salt	Green flashes seen with naked eye	Zn^{2+} , Mn^{2+} may be present

with cone. HCl and performed flame test.		
6. Borax bead test Did not perform this test since the given salt was white.	–	Cu^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}, Fe^{3+} may be absent.
7. Dil. sulphuric acid test Treated a pinch of the salt with dil. H_2SO_4 and warmed. Shook a pinch of salt with water taken in test tube.	Colourless, odourless gas evolved with brisk effervescence, turned lime water milky. Salt did not dissolve.	CO_3^{2-} present Insoluble CO_3^{2-} indicated.
8. $KMnO_4$ test To a pinch of the salt added dil. H_2SO_4 and a drop of $KMnO_4$ solution.	Pink colour of $KMnO_4$ was not discharged.	Cl^-, Br^-, I^-, $C_2O_4^{2-}$ and Fe^{2+} be absent.
9. Cone, sulphuric acid test This test was not performed as the salt reacted with dil. H_2SO_4.	–	Cl^-, Br^-, I^-, $C_2O_4^{2-}$, CH_3COO^- and Fe^{2+} may be absent.
10. Confirmatory test for carbonate	The salt is insoluble in water.	Insoluble carbonate.

Tried to dissolve the salt in water. To the salt added dil HCl	Brisk effervescence with evolution of colourless, odourless gas which turned lime water milky.	Insoluble carbonate confirmed.

Result

Acid radical : CO_3^{2-}
Basic radical : Zn^{2+}