GOVT. POLYTECHNIC, GAJAPATI

Experiment No. : 09 Page No. : 37 Date :

AIM OF THE EXPERIMENT :-

To test the unknown Basic and Acid radical present in the supplied salt.

APPARATUS REQUIRED :-

1. Test tubes 2. Test tube holder 3. Watch glass 4. Blow pipe 5. Nichrome wire 6. Blue glass 7. Charcoal cavity

CHEMICALS REQUIRED :-

1. Given salt 2. Various reagent 3. Litmus paper

THEORY AND PROCEDURE :-

1. PRELIMINARY TEST :

(a) Salt No. :

(b) Colour of the salt :

(c) Structure of the salt :

(d) Solubility :

2. DRY TEST FOR BASIC RADICALS :-

Dry Test tube Heating :

EXPERIMENT	OBSERVATION	INFERENCE
A small quantity of salt was taken in a clean and dry test tube and heated strongly in the hottest part of the non-luminous flame.	(a) A sublimate was formed. (Note the colour of the salt)	(a) It is volatile salt. (Sodaline test and bulb tube test should be performed.)
	(b) Water particles condensed at the cooler part of the test tube.	(b) Salt contains water of crystallisation.
	(c) Decrepitation or cracking sound was produced.	(c) May be crystalline salt.
	(d) Deflagration took place	(d) The salt may be nitrate of alkali or alkaline earth metal.
	(e) The salt changed its colour Yellow when hot and white when cold.	(e) It may be Zinc salt.

	(f) Salt fused on heating and solidified on cooling.	(f) May be alkali or alkaline earth metal salt.

3. SODALIME TEST :-

EXPERIMENT	OBSERVATION	INFERENCE
A little of the salt was taken in a clean watch glass along with soda-lime and it was rubbed by adding two drops of water.	A colourless gas evolved with strong smell of ammonia and colour of the mixture was unchanged.	NH_4^+ may be present. (To be confirmed in the wet test)

4. CHARCOAL CAVITY HEATING (Oxidising flame).

EXPERIMENT	OBSERVATION	INFERENCE
A little of the salt was taken in the charcoal cavity and heated by oxidising flame with the help of a blow pipe.	(a) The salt decrepitated.	(a) May be crystalline salt.
	(b) The salt deflagrated.	(b) May be NO_3^- salt.
	(c) The salt fused and sinked into the charcoal cavity.	(c) Salt contains alkali or alkaline earth metal. (Flame test should be performed)
	(d) Infusible incandescent white residue was obtained	(d) Cobalt nitrate test should be performed.

5. COBALT NITRATE TEST :-

EXPERIMENT	OBSERVATION	INFERENCE
The salt was taken in the charcoal cavity and heated in the oxidizing flame with the help of a blow pipe till an infusible, incandescent white mass was obtained. Then one drop of cobalt nitrate solution was added to it and heated strongly.	(a) Blue mass was obtained.	(a) Al^{3+} may be present.
	(b) Green mass was obtained	(b) Zn^{2+} may be present.
	(c) Rosy mass was obtained.	(c) Mg^{2+} may be present.
	(d) Grey mass was obtained.	(d) Ca^{2+} may be present.

Experiment No. : Page No. : 39 Date :

(6) FLAME TEST :-

EXPERIMENT	OBSERVATION	INFERENCE
The nichrome wire was cleaned with sand paper and dipped in conc. HCl and shown to non-luminous flame. This process was repeated till no colour was imparted to the flame. Then the wire was moistened with conc. HCl and a little of the salt was taken by touching to the salt and shown to the oxidizing flame.	(a) Persistent golden yellow coloured flame was seen in naked eye and colourless through double blue glass.	(a) Na^+ may be present.
	(b) Violet flame was seen in naked eye and red through a pair of blue glass.	(b) K^+ may be present.
	(c) Brick red flame was observed.	(c) Ca^{2+} may be present.

7. IDENTIFICATION OF ACID RADICAL :-

Test for Gr-I Acid Radicals (Carbonate and Sulphide)

EXPERIMENT	OBSERVATION	INFERENCE
1cc dilute HCl was taken in the test tube and slightly warmed. To this a pinch of the supplied salt was added.	(a) Effervescence took place with the evolution of a colourless, odourless gas was evolved.	(a) Carbonate (CO_3^{2-}) may be present (other test should be performed for its confirmation.
	(b) Effervescence took place with the evolution of a colourless, odourless gas with rotten egg smell was evolved.	(b) Sulphide (S^{2-}) may be present (other test should be performed for its confirmation.

Test for Gr-II Acid Radicals (Chloride)

EXPERIMENT	OBSERVATION	INFERENCE
A few drops of conc. H_2SO_4 was taken in a clean and dry test tube, a pinch of the supplied salt was added into it and was gently warmed.	A colourless fuming gas with pungent odour was evolved.	Cl^- may be present. (Other test should be performed for its confirmation.)

Experiment No. : Page No. : 40 Date :

Test for Give-III Acid Radicals (Nitrate and Sulphate) :-

Test for Nitrate (NO_3^-) :

EXPERIMENT	OBSERVATION	INFERENCE
A pinch of the supplied salt was moistened with a few drops of conc. H_2SO_4 taken in a clean and dry test tube and was gently warmed.	A brown fume with pungent smell was observed.	May be NO_3^-. (other test should be performed for its confirmation).

Test for Sulphate (SO_4^{2-})

EXPERIMENT	OBSERVATION	INFERENCE
1-2 cc of the salt solution was taken in a clean test tube and was acidified with dil. HCl. A few few cc of Barium chloride ($BaCl_2$) solution was added into it.	A white ppt. was obtained which was soluble in conc. HCl even on boiling	SO_4^{2-} Confirmed.

Confirmatory Tests for Carbonate (CO_3^{2-}).

EXPERIMENT	OBSERVATION	INFERENCE
(1) A burning match stick was shown to the evolved gas.	(1) The burning stick extinguished.	(1) CO_3^{2-} may be present.
(2) A little more salt was added to the above test tube and the evolved gas was passed through	(2) At first white turbidity (milk colour) appeared which disappeared with excess passing of the gas.	(2) CO_3^{2-} may be present.
(3) A little more salt was added to the above test tube and the evolved gas was passed through acidified Potassium dichromate solution with the help of a delivery tube	(3) No change of the colour took place	(3) CO_3^{2-} Confirmed

Experiment No. : Page No. : 41 Date :

Confirematorey Test fore Sulphide (S^{2-}) :-

EXPERIMENT	OBSERVATION	INFERENCE
A filtere papere soaked with Lead acetate solution was shown to the mouth of the test tube.	The filtere papere turened black.	S^{2-} confiremed.

Confirematorey Test fore Chloreide (Cl^-) :-

EXPERIMENT	OBSERVATION	INFERENCE
(1) A glass rood dipped in conc. NH_4OH solution was shown to the gas evolved.	(1) A white dense fume was foremed.	(1) $Cl^\ominus$ may be present.
(2) A pinch of MnO_2 was added to the above test tube and was waremed gently.	(2) A greenish yellow gas was foremed which turened starch iodide papere blue.	(2) $Cl^\ominus$ may be present.
(3) A pinch of the given salt was taken in a clean and drey test tube and was acidified with dil. HNO_3 solution. And a few droops of silvere nitreate ($AgNO_3$) solution was added into it.	(3) A curedy white ppt. was foremed which was soluble in dil. NH_4OH and was soluble in dil. HNO_3	(3) $Cl^\ominus$ confiremed.

Confirematorey Test fore Nitreate (NO_3^-) :-

EXPERIMENT	OBSERVATION	INFERENCE
(1) A pinch of the supplied salt and a few coppere turenings were taken in a clean test tube. 1-2 cc of 50% conc. H_2SO_4 was added into it and was heated gently.	(1) Deep brown vapours were foremed and the solution turned bluish green or green.	(1) May be NO_3^-.
(2) A piece of filtere papere soaked in $FeSO_4$ solution was shown to the evolved gas.	(2) It turned black.	(2) May be NO_3^-.
(3) 1 cc of the supplied salt solution	(3) A brown ring was foremed	NO_3^- confiremed.

in water was taken in a clean test tube. Equal volume of conc. H_2SO_4 was added into the test tube. The test tube was cooled under tap water. And equal volume of freshly prepared ferrous sulphate ($FeSO_4$) solution was added from the side of the test tube.	at the junction of the two liquids. The ring disappeared on shaking.	

8. WET TESTS FOR BASIC RADICALS (Group Analysis)

EXPERIMENT	OBSERVATION	INFERENCE
(1) To 1ml of salt solution in a clean test tube, 1cc of dil. HCl was added.	(a) A white precipitate was formed.	(a) One of the Gr. I basic radicals (Pb^{2+}, Ag^{+}, Hg_2^{2+}) may be present. (Analysis of Gr. I basic radicals should be performed)
	(b) No white precipitate was formed.	(b) Gr. I basic radicals are absent.
(2) To 1ml of the salt solution in a clean test tube, solid NH_4Cl was added till saturation followed by addition of dil. NH_4OH till alkaline.	(a) A precipitate was obtained. (Colour should be noted)	(a) One of the Gr III A basic radicals (Fe^{3+}, Al^{3+}, Cr^{3+}) may be present (Analysis of Gr III A basic radicals should be performed).
	(b) No precipitate was formed.	(b) Gr III A basic radicals are absent.
(3) Through the contents of the above test tube H_2S gas was passed under pressure.	(a) precipitate was formed (colour should be noted).	(a) One of the Gr III B basic radicals radicals (Zn^{2+}, Mn^{2+} Co^{2+}, Ni^{2+}) may be present (analysis of Gr III B radicals should be performed).

	(b) No precipitate was formed.	(b) Gre IIIB basic readicals are absent.
(4) To 1cc of the salt solution was taken in a clean test tube, solid NH_4Cl was added till saturation followed by addition of dil. NH_4OH till alkaline. To this saturated solution of ammonium carbonate was added.	(a) Precipitate was formed. (Colour should be noted).	(a) One of the Gre. IV basic readicals (Ba^{2+}, Sr^{2+}, Ca^{2+}) may be present (analysis of Gre IV readicals should be performed).
	(b) No precipitate was formed.	(b) Gre IV basic readicals are absent.

The above basic readicals are absent indicating that one of the Gre. V basic readicals may be present. As there is no specific group reagent for Gre V, test for individual readicals should be performed.

9. ANALYSIS OF BASIC RADICALS (Group Wise) :-

Analysis of Gre. IIIA Basic Radicals (Al^{3+}) :

EXPERIMENT	OBSERVATION	INFERENCE
(1) 1-2 cc of the supplied salt solution was saturated with solid NH_4Cl followed by the addition of dil. NH_4OH solution till alkaline.	(1) A white ppt. was formed.	(1) It may be Al^{3+}.
(2) 1-2 cc of the supplied salt solution was treated with dil. NaOH solution drop wise and then in excess.	(2) A white ppt. of $Al(OH)_3$ was formed which dissolved in excess of the reagent.	(2) May be Al^{3+}.
(3) 1 cc of the supplied salt solution, disodium hydrogen phosphate was added.	(3) A gelatinous white ppt. of $AlPO_4$ was formed which was soluble in dil. HCl solution.	Al^{3+} confirmed.

Experiment No. : Page No. : 44 Date :

Analysis of Gre. IIIB Basic Radicals (Zn^{2+}) :-

EXPERIMENT	OBSERVATION	INFERENCE
(1) 1-2 cc of the supplied salt solution was saturated with solid NH_4Cl followed by the addition of dil. NH_4OH solution till alkaline. Then H_2S gas was passed through it.	(1) A white ppt. was formed.	(1) May be Zn^{2+}.
(2) 1-2 cc of the supplied salt solution was treated with potassium ferrocyanide solution drop by drop and then in excess.	(2) A white ppt. was obtained.	(2) May be Zn^{2+}.
(3) Dil. NaOH solution was added to 1 cc of the salt solution drop by drop and then in excess.	(3) A gelatinous white ppt. was formed which is soluble in excess of NaOH solution.	(3) Zn^{2+} confirmed.

Analysis of Gre. IV Basic Radicals (Ca^{2+}) :-

EXPERIMENT	OBSERVATION	INFERENCE
(1) 1-2 cc of the supplied salt solution was saturated with solid NH_4Cl and then made alkaline with dil. NH_4OH solution. Then saturated solution of ammonium carbonate $[(NH_4)_2CO_3]$ was added.	(1) A white ppt. of $CaCO_3$ was formed.	(1) May be Ca^{2+}.
(2) The above ppt. was dissolved in a minimum quantity of dil. CH_3COOH. The solution was boiled to remove CO_2 and then ammonium oxalate solution was added to it.	(2) A white ppt. of CaC_2O_4 was formed which was soluble in dil. HCl but insoluble in CH_3COOH.	(2) May be Ca^{2+}.
(3) Flame test was performed with the white ppt. formed above.	(3) Brick red flame was noticed.	(3) Ca^{2+} confirmed.

Experiment No. : Page No. : 45 Date :

Analysis of Gr. V Basic Radicals (NH_4^+, Na^+, K^+) :-

Test for NH_4^+ :

EXPERIMENT	OBSERVATION	INFERENCE
(1) A small quantity of the salt was treated with sodalime and two drops of water and then the mixture was rubbed in a mortar.	(1) A colourless gas having smell of ammonia which produced dense white fumes with a glass rod dipped in conc. NH_4OH. There was no change in the colour of the residue	(1) May be NH_4^+
(2) Nessler's reagent was added to 1cc of the salt solution.	(2) A brown ppt. was obtained.	(2) NH_4^+ confirmed.

Test for Na^+ :-

EXPERIMENT	OBSERVATION	INFERENCE
Potassium Pyroantimonate solution was added to 1cc of the supplied salt solution.	A white crystalline ppt. was formed.	Na^+ confirmed.

Test for K^+ :-

EXPERIMENT	OBSERVATION	INFERENCE
1cc of the salt solution was treated with two drops of cobalt nitrate solution followed by the addition of solid $NaNO_2$ and dil. CH_3COOH solution.	A yellow ppt. was formed.	K^+ confirmed.

CONCLUSION :- Hence, the basic part of the supplied salt is ____ and the acid part of the salt is ____. Thus, the salt supplied is ____.