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## AIM OF THE EXPERIMENT :-

To test the unknown basic radicals present in the given salt.

## APPARATUS REQUIRED :-

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

## CHEMICALS REQUIRED :-

1. Given salt
2. Various reagents
3. Litmus papers

## THEORY AND PROCEDURE :-

### Preliminary Test :

1. Salt No. :
2. Colour of the salt :
3. Structure :
4. Solubility of the salt :

### Dry Test for Basic Radicals :-

1. Dry test tube heating :

EXPERIMENT	OBSERVATION	INFERENCE
A pinch of the salt was taken in a clean and dry test tube and heated.	(a) Water vapours condensed at the cooler part of the test tube. (b) Decrepitation took place. (c) Salt was volatilised and white sublimate was formed. (d) Salt was first melted and finally infusible white mass left. (e) Salt was fused on heating and solidified on cooling. (f) The colour of the salt was changed. (i) Yellow when hot and white when cold. (ii) Yellow when hot and cold.	(a) Salt with water of crystallisation. (b) May be crystalline salt. (c) May be Volatile salt of $NH_4^+$ , $As^{3+}$ and $Hg^{2+}$ . (d) May be $Mg^{2+}$ , $Al^{3+}$ , $Zn^{2+}$ , $Ba^{2+}$ , $Ca^{2+}$ , $Sr^{2+}$ etc. (e) May be alkali or alkaline earth metal. (f) May be salt of $Pb^{2+}$ , $Bi^{2+}$ , $Sn^{2+}$ etc. The salt is non-volatile. (i) May be $Zn^{2+}$ salt. (ii) May be $Pb^{2+}$ salt.

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- (iii) Yellowish brown in hot and yellow when cold. (iii) May be  $\text{Sn}^{2+}$ , or  $\text{Bi}^{3+}$  salt.
- (iv) Black residue (iv) May be  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Mn}^{2+}$  or  $\text{Fe}^{2+}$  salt.

## II. Heating in a Charcoal cavity :-

EXPERIMENT	OBSERVATION	INFERENCE
A pinch of the salt was taken in a charcoal cavity and was heated in oxidising flame with a blow pipe.	(a) Salt was completely volatilised. (b) An infusible incandescent white mass was obtained. (c) The salt was fused and sank into the charcoal cavity and reappeared on cooling. (d) Original salt was white and formed a coloured mass. (e) Original salt was coloured and formed a coloured mass.	(a) May be salt of $\text{NH}_4^+$ , $\text{As}^{3+}$ and $\text{Hg}^{2+}$ . (Sodalime test is to be performed) (b) May be $\text{Mg}^{2+}$ , $\text{Al}^{3+}$ , $\text{Zn}^{2+}$ , $\text{Ba}^{2+}$ , $\text{Ca}^{2+}$ , $\text{Sr}^{2+}$ , $\text{Sn}^{2+}$ etc. (Cobalt nitrate test is to be performed). (c) May be alkali or alkaline earth metal salt. (Flame test is to be performed). (d) May be salt of $\text{Pb}^{2+}$ , $\text{Bi}^{2+}$ , $\text{Sn}^{2+}$ , $\text{Ag}^+$ etc. (Reduction test is to be performed). (e) May be $\text{Cr}^{3+}$ , $\text{Ag}^+$ , $\text{Mn}^{2+}$ etc. (Borax bead test is to be performed.)

## III. Sodalime Test (For Volatile Salt) :-

EXPERIMENT	OBSERVATION	INFERENCE
A pinch of salt was taken in a watch glass.	(a) Ammonia gas was evolved and the colour of the mixture was not changed. (b) Only colour of the residue was changed to brown and there was no evolution of gas.	(a) May be $\text{NH}_4^+$ . (b) May be $\text{Hg}^{2+}$
A little soda lime was added with a drop of water. Then it was rubbed.	(c) No gas was evolved and no change in colour of residue.	(c) May be $\text{As}^{3+}$ .

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## IV. Bulb tube test (For Volatile Salt)

EXPERIMENT	OBSERVATION	INFERENCE
A mixture of salt, anhydrous $\text{Na}_2\text{CO}_3$ and charcoal powder in the proportion 1:3:1 was prepared. A little of the mixture was taken in a bulb tube and heated.	(i) A white shining mirror was formed (ii) A black shining mirror was formed with the evolution of a gas having garlic colour.	(i) May be $\text{Hg}^{2+}$ . (ii) May be $\text{As}^{3+}$ .

## V. Cobalt Nitrate test (For infusible salt) :-

EXPERIMENT	OBSERVATION	INFERENCE
A pinch of salt was taken in a charcoal cavity. It was heated in an oxidising flame till an infusible mass was obtained. A drop of cobalt nitrate solution was added and again heated strongly.	(i) Blue mass (ii) Green mass (iii) Pink mass (iv) Grey mass	(i) May be $\text{Al}^{3+}$ . (ii) May be $\text{Zn}^{2+}$ . (iii) May be $\text{Mg}^{2+}$ . (iv) May be $\text{Ca}^{2+}$ . (Flame test to be performed).

## VI. Flame test. (For fusible salt)

EXPERIMENT	OBSERVATION	INFERENCE
A clean nichrome wire was moistened with concentrated HCl and touched it with a little of the powdered salt. It was shown to the non-luminous flame. The colour of the flame was observed in naked eye and through double blue glass.	Colour through naked eye Colour through double blue glass (i) Golden yellow (ii) Violet (iii) Brick red	(i) Colourless (ii) Red (iii) Light yellow (i) May be $\text{Na}^+$ . (ii) May be $\text{K}^+$ (iii) May be $\text{Ca}^{2+}$

## VII. Charcoal Reduction test. (For white salt changing colour).

EXPERIMENT	OBSERVATION	INFERENCE
A mixture of salt and fusion mixture in the proportion of 1:1	(i) White shining malleable bead without incrustation which	(i) May be $\text{Ag}^+$

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was prepared. A little of this mixture was taken in charcoal cavity and was heated in a reducing flame. did not mark on paper.  
 (ii) White shining malleable bead with lemon yellow incrustation which marked on paper.  
 (iii) May be  $Pb^{2+}$

Wet test for Basic Radicals :-

## 1. Group Analysis :

EXPERIMENT	OBSERVATION	INFERENCE
(1) To 1ml of salt solution was taken 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 supplied 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.	(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) A precipitate was formed (Colour should be noted)	(a) One of the Gr. III B basic 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) Gr. III B basic radicals are absent.
(4) To 1cc of the salt solution was taken in a	(a) Precipitate was formed (colour should be noted).	(a) One of the Gr. IV basic radicals ( $Ba^{2+}, Sr^{2+}, Ca^{2+}$ ) may

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test tube, solid  $\text{NH}_4\text{Cl}$  was added till saturation followed by addition of dil.  $\text{NH}_4\text{OH}$  till alkaline. To this, saturated solution of ammonium carbonate was added.

(b) No precipitate was formed.

be present (analysis of Gr. IV radicals should be performed).  
(b) Gr. IV basic radicals are absent.

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

## 2. Analysis of Basic Radicals (Group Wise) :-

### (i) Analysis of Gr. IIIA Basic Radicals ( $\text{Al}^{3+}$ ):

EXPERIMENT	OBSERVATION	INFERENCE
(1) 1-2 cc of the supplied salt solution was saturated with solid $\text{NH}_4\text{Cl}$ followed by the addition of dil. $\text{NH}_4\text{OH}$ solution till alkaline.	A white ppt. was formed.	May be $\text{Al}^{3+}$ .
(2) 1-2 cc of the supplied salt solution was treated with dil. $\text{NaOH}$ solution dropwise and then in excess.	A white ppt. of $\text{Al}(\text{OH})_3$ was formed which dissolved in excess of the reagent.	May be $\text{Al}^{3+}$ .
(3) 1 cc of the supplied salt solution, disodium hydrogen phosphate was added.	A gelatinous white ppt. of $\text{AlPO}_4$ was formed which was soluble in dil. $\text{HCl}$ solution.	$\text{Al}^{3+}$ is confirmed.

### (ii) Analysis of Gr. IIIB Basic Radicals ( $\text{Zn}^{2+}$ ):

EXPERIMENT	OBSERVATION	INFERENCE
(1) 1-2 cc of the supplied salt solution was saturated with solid $\text{NH}_4\text{Cl}$ followed by the addition of dil. $\text{NH}_4\text{OH}$ solution till alkaline. Then $\text{H}_2\text{S}$ gas was passed through it.	A white ppt. was formed.	May be $\text{Zn}^{2+}$ .
(2) 1-2 cc of the supplied salt solution was treated with potassium ferrocyanide	A white ppt. was obtained.	May be $\text{Zn}^{2+}$ .

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solution drop by drop and then in excess.

(3) Dil. NaOH solution was added to 1 cc of the salt solution drop by drop and then in excess.

A gelatinous white ppt. was formed which was soluble in excess of NaOH solution.

$Zn^{2+}$  is confirmed.

### (iii) Analysis of Gr. 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.

A white ppt. of  $CaCO_3$  was formed.

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.

A white ppt. of  $CaC_2O_4$  was formed which was soluble in dil.  $HCl$  but insoluble in  $CH_3COOH$ .

May be  $Ca^{2+}$ .

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

#### EXPERIMENT

#### OBSERVATION

#### INFERENCE

Test for  $NH_4^+$  :

(1) A small quantity of the salt was treated with soda lime and two drops of water and then the mixture was rubbed in a mortar.

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.

May be  $NH_4^+$ .

(2) Nessler's reagent was added to 1 cc of the salt solution.

A brown ppt. was obtained.

$NH_4^+$  is confirmed.

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Test for  $\text{Na}^+$ :-

EXPERIMENT	OBSERVATION	INFERENCE
Potassium pyroantimonate solution was added to 1cc of the supplied salt solution.	A white crystalline ppt. was formed.	$\text{Na}^+$ is confirmed.

Test for  $\text{K}^+$ :-

EXPERIMENT	OBSERVATION	INFERENCE
1 cc of the salt solution was treated with two drops of cobalt nitrate solution followed by the addition of solid $\text{NaNO}_2$ and dil. $\text{CH}_3\text{COOH}$ solution.	A yellow ppt. was formed.	$\text{K}^+$ is confirmed.

CONCLUSION :-

Hence, the basic part of the supplied salt is