KENDRIYA VIDYALAYA

SUBJECT – CHEMISTRY

PROJECT – TO DETECT THE PRESENCE OF RADICAL ACIDIC IN GIVEN SALT.

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THEORY-

Identification Of Acid Radicals (Anions)

The identification of the acid radicals is first done on the basis of preliminary tests. Dry heating test is one of the preliminary tests performed earlier which may give some important information about the acid radical present. The other preliminary tests are based upon the fact that:

1. CO_3^{2-}, S^{2-}, $SO_3^{2-}$and NO_2^- react with dil. H_2S0_4 to give out CO_2, H_2S, SO_2 and NO_2 gas respectively which can be identified by certain tests.

2. Cl^-, Br^-, I^-, NO_3^-, $C_2O_4^{2-}$ and CH_3COO^- react with cone. H_2S0_4 but not with dil. H_2S0_4 to produce characteristic gases.

3. S042- and P043- react neither with dil. H_2S0_4 nor with cone. H_2S0_4. These are therefore, identified by individual tests.

Thus, the acid radicals may be identified by performing the following tests in the order given below :

(i) Dil. H_2S0_4 test. Treat a pinch of the salt with dil. H_2S0_4 and identify the gas evolved.

(ii) Cone. H_2S0_4 test. If no action takes place with dil. H_2S0_4, warm a pinch of the salt with cone. H_2S0_4 and identify the gas evolved.

(iii) Independent Group. (SO_4^{2-} and PO_4^{3-}). If the salt does not react with dil. H_2S0_4 as well as with cone. H_2S0_4, test for SO_4^{2-} and PO_4^{3-} by performing their individual tests.

Let us now discuss these tests in detail one by one.

Dilute Sulphuric Acid Test-

Take a small quantity of the salt in a test-tube and add 1-2 ml of dilute sulphuric acid. Observe whether some gas is evolved or not. If some gas is evolved, identify the gas and draw inferences from Table .

S.NO	OBSERVATION	INFERENCE	
		GAS EVOLVED	POSSIBLE RADICAL
1	Colourless, odourless gas with brisk effervescence, turns lime water milky.	CO_2	CO_3^{2}
2	Colourless gas, smell like that of rotten eggs, turns ' lead acetate paper black.	H_2S	S^{2-}
3	Colourless gas, smell like that of burning sulphur, turns acidified potassium dichromate paper green.	SO_2	SO_3^{2-}
4	Reddish brown gas, pungent smell, turns ferrous sulphate solution black.	NO_2	NO_{2^-}
5	No gas is evolve	----- ----	CO_3^{2-}, S^{2-}, SO_3^{2-} , NO_2^- absent

Note:

1. Do not treat the salt with a large quantity of dilute acid.

2. Do not heat the salt with dilute acid.

Chemical Reactions Involved in Dil. H_2SO_4 Test-

Dilute H_2S0_4 (or dilute HCl) decomposes carbonates, sulphides sulphites and nitrites in cold to and liberates different gases. These gases on identification indicate the nature of the acid radical present in the salt.

1. Carbonates. On treating the solid carbonate, CO_2 is given off in the cold with brisk effervescence.

$$CaCO_3 + H_2SO_4 \longrightarrow CaSO_4 + H_2O + CO_2 \uparrow$$

2. Sulphides. Sulphides when treated with dil. H_2S0_4 give H_2S gas.

$$ZnS + H_2SO_4 \longrightarrow ZnSO_4 + H_2S \uparrow$$

Sulphites. Sulphites when treated with dil. H_2S0_4 give SO_2 gas.

$$Na_2SO_3 + H_2SO_4 \longrightarrow Na_2SO_4 + SO_2 \uparrow + H_2O.$$

4. Nitrites. On treating the solid nitrite with dil. H_2S0_4, nitric oxide (NO) gas is evolved which readily gives dense brown fumes of NO_2 with oxygen of the air.

$$KNO_2 + H_2SO_4 \longrightarrow KHSO_4 + HNO_2 \,] \times 3$$
$$3HNO_2 \longrightarrow HNO_3 + H_2O + 2NO$$
$$3KNO_2 + 3H_2SO_4 \longrightarrow 3KHSO_4 + HNO_3 + H_2O + 2NO$$
$$2NO + O_2 \longrightarrow 2NO_2$$

Colourless → Brown fumes

Potassium Permanganate Test-

To a pinch of salt in test tube add about 2 ml of dilute sulphuric acid. Boil off any gas evolved, add little more of dilute acid and then potassium permanganate solution d'ropwise. Note the changes as given in Table . This test helps in detection of Cl^-, Br^-, I^-, $C_2O_4^{2-}$, and Fe^{2+} radicals.

S.NO	OBSERVATION	INFERANCE
1	Potassium permanganate decolourised without the evolution of any gas.	Presence of Fe^{2+} salts.
2	Potassium permanganate decolourised : (a) In cold (i) With the evolution of chlorine. (ii) With the evolution of bromine. (iii) With the evolution of iodine. (b) On warming (i) With the evolution of carbon dioxide	Cl^- Br^- I^- $C_2O_4^{2-}$
3	$KMnO_4$ not decolourised.	Absence of Cl^-, Br^-, I^-, $C_2O_4^{2-}$ and Fe^{2+}

Note:

Do not perform this test if the salt reacts with dilute sulphuric acid because sulphides, sulphites and nitrites also decolourise KMn04 solution.

Chemical Reactions Involved

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

1. *Ferrous salts* :

$$2FeSO_4 + H_2SO_4 + [O] \longrightarrow Fe_2(SO_4)_3 + H_2O$$

2. *Chlorides* :

$$NaCl + H_2SO_4 \longrightarrow NaHSO_4 + HCl$$
$$2HCl + [O] \longrightarrow H_2O + Cl_2 \uparrow$$

3. *Bromides* :

$$NaBr + H_2SO_4 \longrightarrow NaHSO_4 + HBr$$
$$2HBr + [O] \longrightarrow H_2O + Br_2 \uparrow$$

4. *Iodides* :

$$NaI + H_2SO_4 \longrightarrow NaHSO_4 + HI$$
$$2HI + [O] \longrightarrow H_2O + I_2 \uparrow$$

5. *Oxalates* :

$$\begin{matrix}COONa \\ | \\ COONa\end{matrix} + H_2SO_4 \longrightarrow \begin{matrix}COOH \\ | \\ COOH\end{matrix} + Na_2SO_4$$

$$\begin{matrix}COOH \\ | \\ COOH\end{matrix} + [O] \longrightarrow H_2O + 2CO_2 \uparrow$$

Concentrated Sulphuric Acid Test-

This test is performed by treating small quantity of the salt with cone, sulphuric acid (2-3 ml) in a test tube. Identify the gas evolved in cold and then on heating. Draw inferences from Table . Cone. Sulphuric Acid Test.

S.NO	OBSERVATION	GAS EVOLVED	POSSIBLE RADICALS
1	Colourless gas with pungent smell. The gas gives white fumes with aqueous ammonia (NH_4OH) and white ppt. with $AgNO_3$ solution.	HCL	Cl^-
2	Reddish brown vapours with pungent smell, turns starch paper yellow. It does not turn $FeSO_4$ solution black.	Br_2	Br^-
3	Deep violet vapours with pungent smell, turns starch paper blue. A sublimate is formed on the sides of the tube.	I_2 vapours	I^-

4	**Reddish brown gas with pungent smell, turns $FeSO_4$ solution black.**	**NO_2**	NO_2^-
5	**Colourless gas which turns lime water milky and a gas which burns with blue flame.**	**CO_2 and CO**	$C_2O_4^{2-}$
6	**No gas/vapours evolved.**		**Cl^-, Br^-, I^-, CH_3COO^-, $C_2O_4^{2-}$ absent**

Note :

1. If some gas is evolved with dilute sulphuric acid, then there is no need for performing cone, sulphuric acid test.

2. Do not boil the salt with cone, sulphuric acid. On boiling, the acid may decompose to give SO_2 gas.

3. Nitrates give vapours of nitric acid (colourless) when heated with cone, sulphuric acid. When a paper pellet or copper chips is added, dense brown fumes evolve. Paper pellet acts as a reducing agent and reduces nitric acid to NO_2 (Reddish brown gas).

Chemical Reactions Involved in cone. H_2S0_4 Test

1. *Chlorides*

$$NaCl + H_2SO_4 \longrightarrow NaHSO_4 + HCl$$

Sod. hydrogen sulphate

2. *Bromides*

$$NaBr + H_2SO_4 \longrightarrow NaHSO_4 + HBr$$

$$H_2SO_4 + 2HBr \longrightarrow SO_2 + Br_2 + 2H_2O$$

3. *Iodides*

$$KI + H_2SO_4 \longrightarrow KHSO_4 + HI$$

$$H_2SO_4 + 2HI \longrightarrow SO_2 + I_2 + 2H_2O$$

4. *Nitrates*

$$KNO_3 + H_2SO_4 \longrightarrow KHSO_4 + HNO_3$$

$$4HNO_3 + \underset{\text{(Paper pellet)}}{C} \longrightarrow 4NO_2\uparrow + CO_2 + 2H_2O$$

5. *Acetates*

$$CH_3COONa + H_2SO_4 \longrightarrow NaHSO_4 + \underset{\text{Acetic acid}}{CH_3COOH}$$

6. *Oxalates* :

$$\begin{matrix}COONa\\|\\COONa\end{matrix} + H_2SO_4 \longrightarrow \begin{matrix}COOH\\|\\COOH\end{matrix} + Na_2SO_4$$

$$\begin{matrix}COOH\\|\\COOH\end{matrix} \xrightarrow{H_2SO_4} CO_2\uparrow + CO\uparrow + H_2O$$

Tests For Independent Radicals (SO_4^{2-} and PO_4^{3-})

As already discussed these radicals are not detected by dilute or concentrated H_2SO_4. They are tested individually.

1. SULPHATE (SO_4^{2-})

Boil a small amount of salt with dilute HCl in a test tube. Filter the contents, and to the filtrate add few drops of BaCl2 solution. A white precipitate, insoluble in cone. HCl, indicates presence of sulphate.

2. PHOSPHATE (PO_4^{3-})

Add cone. HNO_3 to the salt in a test tube. Boil the contents and add excess of ammonium molybdate solution. A yellow precipitate indicates presence of phosphate.

S.NO	EXPERIMENT	OBSERVATION	INFERENCE
1	PHYSICAL EXAMINATION Colour of the sample is noted	White colour	Cu_2^+,Fe_2^+,Fe_3^+,Ni_2^+, Mn_2^+,Co_2^+, are absent
2	A little amount of the sample is rubbed between thumb and finger.	Pungent smell evolved	NH_4^+ may be present
3	Dry Test Tube Heating : Heated small amount of the sample in a dry test tube.	Pungent gas evolved along with formation of white sublimate	NH_4^+ ,Cl^-may be present
4	Flame test was performed	No characteristic colour in the flame	Ca_2^+ ,Ba^{2+} Cu^{2+} may be absent.
5	Charcoal activity test was performed.	No characteristic bead but white incrustation around the edge of cavity.	Pb^{2+}, Cu^{2+},Zn^{2+},As^{3+} may be absent and NH^{4+} may be present
6	Small amount of the sample is treated with dilute H_2SO_4 and and pass the gas through lime wate	Effervescence with evolution of colourless, odourless gas	S^{2-}, NO^{2-},may be absent. CO_3^{2-} may be present.
7	Small amount of the solid sample is treated with	Colourless and odourless gas evolved with effervescence	NO^{3-}, Cl^-, Br^- be absent. Co_3^{2-} be present.

	concentrated H_2SO_4		
8	**Small amount of the solid sample is treated with Dilute H_2SO_4 and the evolved gas is passed through lime water.**	**Effervescence with evolution of colourless, odourless gas which turns lime water milky.**	**Co_3^{2-} is present and confirmed.**
	Preparation of aqueous extract : Aqueous extract of the sample was prepared and following test for acid radical was performed.		
9	**Small amount of the sample is treated with dilute H_2SO_4 and and pass the gas through lime water**	**Lime water turns milky and milkiness disappears on passing excess lime water**	**Co_3^{2-} is present and confirmed**
10	**To the aqueous solution of sample $MgSO_4$ solution was added**	**White precipitate was formed**	**$Co_3^{2-}$is present and confirmed**
11	**Acidified the solution with dilute HNO_3 and $AgNO_3$ solution was added**	**No precipitate formed**	**Cl^- is absent**
12	**Acidified the solution with dilute HCl and $BaCl_2$ solution was added**	**No precipitate formed**	**SO_4^{2-} is absent.**
13	**Freshly prepared $FeSO_4$ solution is added to the sample solution**	**No brown ring was formed**	**NO^{3-} was absent.**

	and 2ml of conc.$H2SO_4$ was added carefully along the wall of the container		
14	**Acidified the solution with conc. HNO_3 and a pinch of ammonium molybdate was added to it.**	**No precipitate is produced**	**PO_4^{3-} is absent**
15	**To the aqueous solution of sample $MgSO_4$ solution was added**	**No precipitate was formed**	**$Co_3^{2-}$is absent**
16	**Acidified the solution with dilute HNO_3 and $AgNO_3$ solution was added.**	**Curdy White precipitate formed which dissoved in excess NH4OH**	**Cl^- is present and confirmed**
17	**Freshly prepared $FeSO_4$ solution is added to the sample solution and 2ml of conc.H_2SO_4 was added carefully along the wall of the container.**	**No brown ring was formed**	**NO^{3-} was absent.**