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Diploma in Pharmacy 1st Year

Pharmaceutical Chemistry

Experiment

To perform the identification test of anions

Aim:

To perform the identification test of anions

Reference :

‘ Dr. Gupta G.D. , Dr. Sharma Shailish , Kaur Baljeet ’ “Practical Manual of Pharmaceutical Chemistry” Published by Nirali Prakashan, Page no 21 - 24

Requirements:

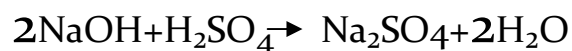
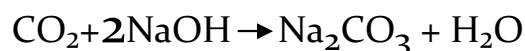
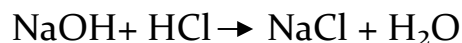
Apparatus Required: Test tube, Test tube holder, spatula, non-luminous bunsen burner, glass rod, con HCl, dilute H₂SO₄, con. H₂SO₄, copper turnings, MnO₂, NaOH solution, dilute HNO₃, sodium carbonate, AgNO₃, solution, BaCl₂ solution, acetic acid.

Material Required: hydrochloric acid, lead acetate solution, FeSO₄, solution, FeCl₃, solution, dil. HCl, calcium chloride, and Na₂CO₃

Theory:

The detection and identification of acidic and basic radicals present in inorganic salts are parts of qualitative analysis Acids and bases, or acidic oxides with a base or basic oxides, react to form inorganic salts.

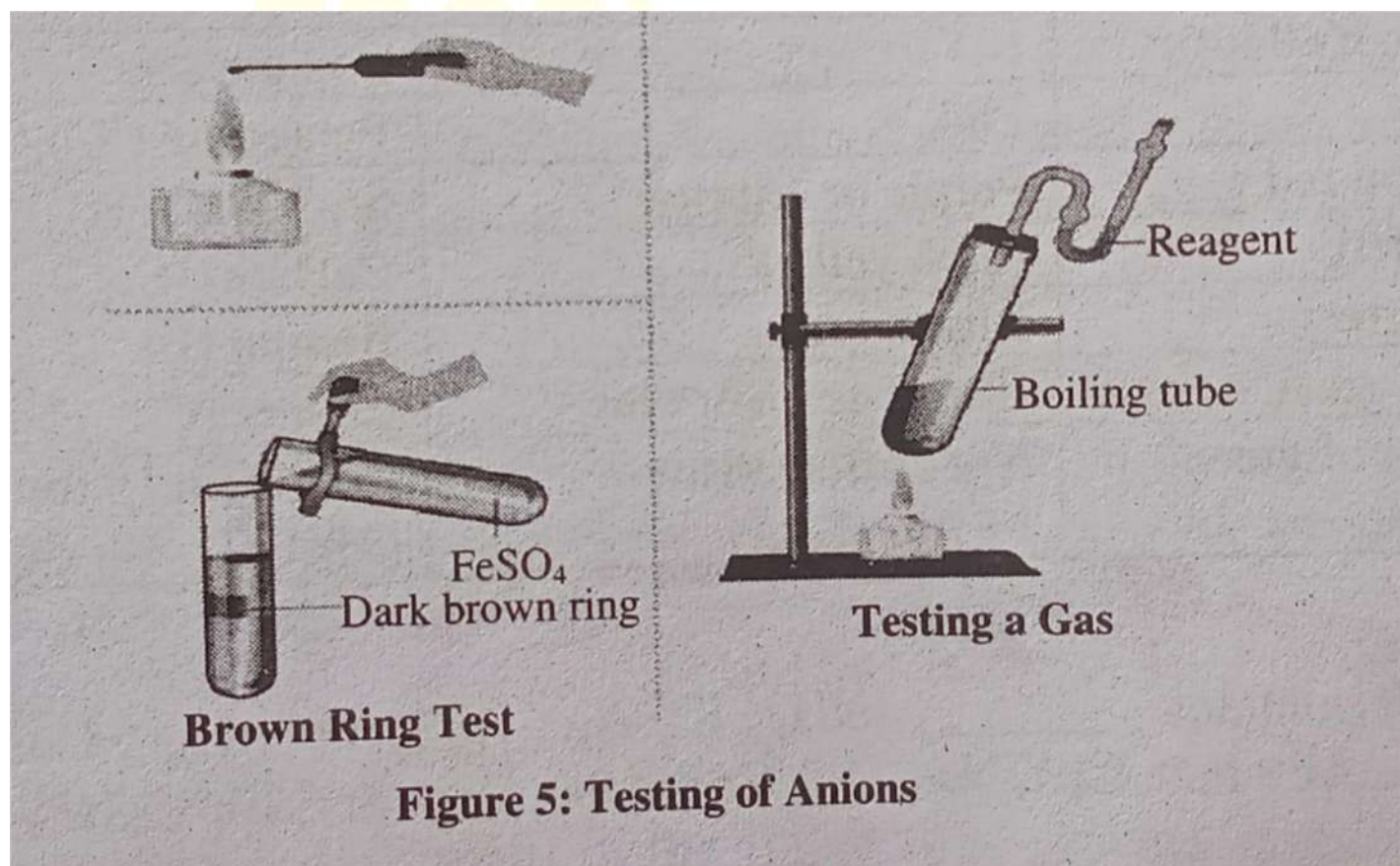
The following are some examples of acids and bases, or acidic oxides reacting with a base or basic oxides:



Because they have fixed geometrical structures, most organic molecules are crystalline solids. They are usually made up of radicals, which are oppositely charged particles or ions.

Two essential principles are extremely useful in salt analysis:

- 1. Solubility Product:** When the solution is saturated, the product of ion concentrations raised to a power equal to the number of occurrences of ions in an equation expressing electrolyte dissociation at a certain temperature is known as solubility product. The solubility product is not the ionic product under every condition, but only when the solution is saturated
- 2. Common Ion Effect:** The common ion effect is a phenomenon in which a small amount of strong electrolyte containing a common ion decreases the degree of dissociation of any weak electrolyte. Ionization of the weak electrolyte acetic acid is controlled, for example, by introducing strong electrolyte sodium acetate containing the common acetate ion.



Procedure

Table 4: Identification Test of Anions

S.No	Experiment	Observation	Inferences Presence of
1)	Preliminary reactions	Given salt is colourless	Absence of Fe^{2+} , Fe^{3+} , Ni^{2+} and Co^{2+} ions.
	Appearance	If the given salt is green	May be Fe^{2+} , Ni^{2+} , Cu^{2+} is Present.
		If the given salt is brown	May be Fe^{2+}
		If the given salt is pink	May be Co^{2+} , Mn^{2+}
		If the given salt is blue	Cu^{2+} is Present.
2)	Action of Heat: In a dry test tube, take a small amount of salt and gently heat it.	A colourless gas with a strong odour that turns moist red litmus paper to become blue.	May be NH_4^+ salt is Present.
		Acidified ferrous sulphate brown paper is turned into reddish brown fumes.	May be NO_3^- is Present.
		The substance is white when cold and yellow when hot.	May be $(\text{Zn})^{2+}$ is Present.
3)	Flame Test: In a watch glass, add a drop of Con.HCl to a small amount of salt and put it in a paste. With the help of a glass rod, place the paste in the base of the non-luminous bunsen burner.	a) Apple green colour flame. b) Crimson red colour flame. c) Brick red colour flame. d) Bluish green flame	May be Ba^{2+} is Present. May be Sr^{2+} is Present. May be Ca^{2+} is Present. May be Cu^{2+} is Present.

3)

4)

Table 5: Identification of Anions from Volatile Products

S.No	Experiments	Observations	Inferences
1)	Action of dilute H_2SO_4: In a test tube, add 1 or 2ccs of dilute H_2SO_4 to a small part of the salt and gently warm it.	Colourless, odourless gas changes lime water into brisk effervescence.	Anion is Carbonate CO_3^{2-} .
		The result is a colourless gas with a rotten egg odour that turns lead acetate black paper.	Presence of sulphide anion.
		The result is a colourless gas with burning sulphur odour that turns acidified dichromate green.	Presence of sulphate anion.
		The result is a colourless gas with a fishy odour that turns acidified brown ferrous sulphate.	Presence of nitrate anion.
		Colorless vinegar-flavoured gas is obtained.	May be acetate anion is present.
		No characteristic observation.	Absence of above mentioned anions.
2)	Action of Conc H_2SO_4: To a small amount of salt in a test tube, add 2-3 ccs of Con. H_2SO_4 and gradually heat it.	Reddish-brown vapours that turn moist red paper fluorescent.	May be bromide anion is present.
		With a dipped glass rod in NH_4OH solution, a colourless gas with a pungent odour produces dense white vapours.	May be chloride anion is present.
		Vapors of violet colour that turn blue, or violet starch paper	May be iodide anion is present.
		Brown ferrous sulphate paper becomes acidified due to reddish-brown fumes.	May be nitrate anion is present.
		No characteristic observation.	Absence of all above-mentioned anions is present.
3)	Action of Conc H_2SO_4 with Cu turnings: In a test tube, mix a small amount of salt with a few Cu pieces, add 2-3 ccs of H_2SO_4 , and heat it.	It is observed that copy evolution of reddish brown gas causes acidified ferrous sulphate paper to turn brown.	Presence of nitrate anion is present.
		No reddish brown vapours.	Absence of nitrate anion.
4)	Action of Conc H_2SO_4 with MnO_2: In a test tube, add an equal amount of MnO_2 with a small amount of salt, then add a few ccs of Con. H_2SO_4 and heat gently.	A greenish yellow gas causes starch iodide paper to turn violet (or) blue.	May be chloride anion is present
		Vapors of a reddish brown colour are produced, which turn moist fluorescent red paper fluorescent.	May be bromide anion is present.
		Violet vapors are obtained that turn starch paper blue (or) violet.	May be iodide anion is present
		No characteristic coloured vapours are obtained.	Absence of all above mentioned anions is present

Sodium Carbonate Extract

The confirmatory tests for CO_3^{2-} , S^{2-} , SO_3^{2-} , NO_2^- , and CH_3COO^- anions:

When the salt is water-soluble, confirmatory anion testing is carried out with water extract and when the salt is water-insoluble, confirmatory anion testing is carried out with sodium carbonate extract. Because carbonate ions are present in the sodium carbonate extract, confirmation of $\text{C} \times \text{O}_{\{3\}}^{\wedge} 2$ is done with an aqueous salt solution or solid salt. Water extract is made by dissolving salt in water.

Preparation of Sodium Carbonate Extract

1 g of salt is taken in a boiling tube or porcelain dish. 3 g of solid sodium carbonate is mixed with 15 ml of distilled water. Cook for about 10 minutes after removing the contents. Cool and then filter the filtrate into a test tube and label it as a sodium carbonate extract.

Table 6: Confirmatory Tests for CO_3^{2-} , S^{2-} , SO_3^{2-} , NO_2^- and CH_3COO^- anions

S.No.	Experiment	Observation	Inference
1)	Silver Nitrate Test: Until the effervescence stops, add dilute HNO_3 to a part of the sodium carbonate extract. Add a few extra drops of AgNO_3 solution, 2–3 nos.	Curdy white NH_4OH soluble precipitate.	May be chloride anion is present.
		Pale yellow precipitate in NH_4OH that is sparingly soluble.	May be bromide anion is present.
		Insoluble yellow precipitation in NH_4OH .	May be iodide anion is present.
		No characteristics precipitate.	Absence of all above mentioned anions.
2)	Barium Chloride Test: Add one or two ccs of BaCl_2 solution to the extract (after acetic acid neutralisation and CO_2 boiling). To a part of the ppt above, add dilute hydrochloric acid.	An insoluble white precipitate in HCl .	The anion SO_4^{2-} is present.
		A HCl soluble white precipitate.	The anion SO_3^{2-} is present.
		No characteristics precipitate.	Absence of SO_4^{2-} and SO_3^{2-} .
3)	Lead Acetate Test: Add one or two ccs of lead acetate solution to the extract (after acidification with acetic acid, CO_2 boiling off and cooling).	White ppt, soluble in excess of the solution of ammonium acetate.	Presence of SO_4^{2-} is confirmed.
4)	Ferrous Sulphate Test (Also called Brown Ring Test): To around 1 or 2cc of extract, add dilute H_2SO_4 in drops until the effervescence ceases.	Brown ring is obtained at the liquid junction.	Presence of nitrate anion (NO_3^-).
		No brown ring formed	Nitrate anion (NO_3^-) is absent.

Observations and Inference

S.No	Anions	Observation
1)	CO_3^{2-} (Carbonate anion)	CO_2 gas is produced with dilute sulphuric acid effervescence, which turns lime water milky.
2)	S^{2-} (Sulphide anion)	Add a drop of sodium nitroprusside solution. The colouration appears to be purple or violet.
3)	SO_3^{2-} (Sulphite anion)	With a barium chloride solution dissolved in weak hydrochloric acid, a white precipitate forms, as well as sulphur dioxide gas.
4)	SO_4^{2-} (Sulphate anion)	After acidification with weak hydrochloric acid, dissolve 1 mL of salt water extract in water or sodium carbonate and add BaCl_2 solution. White insoluble precipitate in concentrated HCl or HCl. It gets HNO_3 .
5)	NO_2^- (Nitrite anion)	Acidify with acetic acid after adding a few drops of iodide potassium solution and a few drops of starch solution. The blue colour appears.
6)	NO_3^- (Nitrate anion)	1 ml of salt solution is taken in water and then add a conc. of 2 ml. H_2SO_4 is completely mixed. The mixture is then cooled under the tap. Freshly prepared ferrous sulphate is added without shaking on the sides of the test tube. A dark brown ring is formed at the junction of two solutions.
7)	Cl^- (Chloride anion)	0.1gm of salt is taken in a test tube and add a pinch of manganese dioxide and 3-4 drops of conc. sulphuric acid. The mixture is then heated. Chlorine gas is a greenish yellow gas with a strong odour and bleaching effect that can be recognised.

8)	Br^- (Bromide anion)	0.1gm of salt is taken in a test tube and add a pinch of manganese dioxide and 3-4 drops of conc. sulphuric acid. Strong brown fumes are produced.
9)	I^- (Iodide anion)	1ml of salt solution is taken in a test tube. Add a conc of 2 mL. H_2SO_4 is completely mixed. The mixture is then cooled under the tap. Freshly prepared ferrous sulphate is added without shaking on the sides of the test tube. A dark brown ring is formed at the junction of two solutions.
10)	PO_4^{3-} (Phosphate anion)	Con HNO_3 and the extract of sodium carbonate or salt solution in water is acidified, then add the ammonium molybdate solution and heat to a boil. The result is a precipitate that is canary yellow in colour.
11)	$\text{C}_2\text{O}_4^{2-}$ (Oxalate anion)	1 mL of acetate acidified water extract or sodium carbonate extract is taken and a solution of calcium chloride is added. In a solution of ammonium oxalate and oxalic acid, an insoluble white precipitate forms, but it is soluble in dilute hydrochloric acid and diluted nitric acid.
12)	CH_3COO^- (Acetate anion)	1 mL and 0.2 mL conc. of ethanol is added. H_2SO_4 is added and then heat it. Fruity odour confirms the presence of acetate ion.

Result: The given salt contains _____ (CO_3^{2-} , S^{2-} , SO_3^{2-} , SO_4^{2-} , NO_2^- , NO_3^- , Cl^- , Br^- , I^- , PO_4^{3-} , CO_4^{2-} , CH_3COO^-) anion.

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Amir Khan

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