Section 2

Identification Tests for Anions and Cations

Experiment 5: Identification of sodium cation and thiosulphate anion in sodium thiosulphate
Experiment 6: Identification of magnesium cation and sulphate anion in magnesium sulphate
Experiment 7: Identification of ammonium cation and chloride anion in ammonium chloride
Experiment 8: Identification of sodium cation and bicarbonate anion in sodium bicarbonate
Experiment 9: Identification of potassium cation and iodide anion in potassium iodide
Experiment 10: Identification of barium cation and sulphate anion in barium sulphate
Experiment 11: Identification of calcium cation and gluconate anion in calcium gluconate
Experiment 12: Identification of potassium cation and citrate anion in potassium citrate

SODIUM THIOSULPHATE ($Na_2S_2O_3$. $5H_2O$)

Category

It is an antidote to cyanide poisoning.

Description

Large colourless crystals or coarse, crystalline powder; odourless; deliquescent in moist air and effloresces in dry air at temperature above 33°C.

Solubility

Very soluble in water; practically insoluble in ethanol.

Identification Tests

Sodium thiosulphate gives reactions for sodium ions and for thiosulphate ions.

Reactions of Sodium Ions

1. Take 2 ml of test solution and add 2 ml of 15% w/v solution of potassium carbonate. Heat to boiling. No precipitate is produced. Add 4 ml of freshly prepared potassium antimonate solution and heat to boiling. Allow to cool in ice and if necessary scratch the inside of the test tube with a glass rod; a dense, white precipitate is formed.

   Sodium ions react with a solution of potassium antimonate ($KSbO_3$, H_2O) to form a white precipitate of sodium antimonate ($NaSbO_3$)

$$\underset{\substack{\text{Potassium}\\\text{antimonate}}}{KSbO_3} + Na^+ \longrightarrow \underset{\substack{\text{Sodium}\\\text{antimonate}\\\text{(white ppt)}}}{NaSbO_3\downarrow} + K^+$$

2. Acidify a solution of the substance being examined with acetic acid and add a large excess of magnesium uranyl acetate solution; a yellow, crystalline precipitate of sodium nagnesium uranyl acetate is formed.

$$Na^+ + Mg^{++} + 3UO^{2--} + 9CH_3COO^- \longrightarrow \underset{\substack{\text{Sodium magnesium}\\\text{uranyl acetate}\\\text{(yellow ppt)}}}{NaMg(UO_2)_3(CH_3COO)_9\downarrow}$$

Reactions of Thiosulphate Ions

Test solution: 10% w/v solution of sodium thiosulphate in carbon dioxide free water.

1. To 0.5 ml of test solution, add 0.5 ml of water and 2 ml of 0.1 M silver nitrate solution. A white precipitate is produced which quickly becomes yellowish and finally black.

Experiment 5: Identification of Sodium Cation and Thiosulphate Anion in Sodium Thiosulphate

Date________________

AIM

To perform the identification tests for the given sample of sodium thiosulphate.

CHEMICALS REQUIRED

Sodium thiosulphate, potassium carbonate, potassium antimonate solution, acetic acid, magnesium uranyl acetate solution, silver nitrate, iodine solution, hydrochloric acid, starch-iodide paper.

APPARATUS REQUIRED

Test tubes, test tube stand, test tube holder, glass rod.

PROCEDURE

The sample gives the reactions of sodium ions and thiosulphate ions.

REACTIONS OF SODIUM IONS

S. no.	*Test*	*Observation*	*Inference*
1.	Dissolve 0. 1 g of the substance in 2 ml of water. Add 2 ml of 15% w/v solution of potassium carbonate and heat to boiling. Add 4 ml of freshly prepared potassium antimonate solution and heat to boiling. Allow to cool in ice and if necessary scratch the inside of the test tube with a glass rod.		
2.	Take 0.1 g of the substance and acidify with 1 M acetic acid. Add a large excess of magnesium uranyl acetate solution.		

REACTIONS OF THIOSULPHATE IONS

S. no.	*Test*	*Observation*	*Inference*
1.	To 0.5 ml of test solution add 0.5 ml of water and 2 ml of 0.1 M silver nitrate solution.		

S. no.	Test	Observation	Inference
2.	To 0.5 ml of the test solution add a few drops of iodine solution.		
3.	To 2.5 ml of test solution add 2.5 ml of water and l ml of hydrochloric acid.		
4.	Dissolve 0.1 g of the sample in 5 ml of water and add 2 ml of ferric chloride test solution.		
5.	To 0.5 ml of test solution add 2 drops of bromine solution.		

RESULT

The sample gives the identification tests for sodium thiosulphate.

MAGNESIUM SULPHATE ($MgSO_4.7H_2O$)

Category

Osmotic laxative; used in the treatment of electrolyte deficiency.

Description

Colourless crystals or white, crystalline powder, odourless.

Solubility

Very soluble in boiling water, freely soluble in water, practically insoluble in ethanol.

Identification Tests

Magnesium sulphate gives reactions of magnesium ions and sulphates.

Reactions of Magnesium

1. Dissolve about 15 mg of the substance being examined in 2 ml of water. Add 1 ml of dilute ammonia solution. A white precipitate forms that is redissolved by adding 1 ml of 2 M ammonium chloride. Add 1 ml of 0.25 M disodium hydrogen phosphate; a white, crystalline precipitate is produced.

$$\underset{\substack{\text{Magnesium}\\ \text{sulphate}}}{MgSO_4} + \underset{\substack{\text{Ammonium}\\ \text{hydroxide}}}{2NH_4OH} \longrightarrow \underset{\substack{\text{Magnesium}\\ \text{hydroxide}\\ \text{white ppt.}}}{Mg(OH)_2\downarrow} + \underset{\substack{\text{Ammonium}\\ \text{sulphate}}}{(NH_4)_2\,SO_4}$$

2. The precipitate of magnesium hydroxide dissolves in ammonium chloride.
3. When we add disodium hydrogen phosphate; white , crystalline precipitate is formed.

$$\underset{\substack{\text{Magnesium}\\ \text{hydroxide}}}{Mg(OH)_2} + \underset{\substack{\text{Disodium}\\ \text{hydrogen}\\ \text{phosphate}}}{Na_2HPO_4} \longrightarrow \underset{\substack{\text{Magnesium ammonium}\\ \text{phosphate (white ppt)}}}{Mg(NH_4)\,PO_4\downarrow}$$

Reactions of Sulphates

1. Dissolve about 50 mg of sample in 5 ml of water. Add 1 ml of dilute hydrochloric acid and 1 ml of barium chloride solution; a white precipitate is formed.

$$\underset{\substack{\text{Magnesium}\\ \text{sulphate}}}{MgSO_4} + \underset{\substack{\text{Barium}\\ \text{chloride}}}{BaCl_2} \longrightarrow \underset{\substack{\text{Magnesium}\\ \text{chloride}}}{MgCl_2} + \underset{\substack{\text{Barium}\\ \text{sulphate}\\ \text{(white ppt)}}}{BaSO_4\downarrow}$$

2. Dissolve about 50 mg of the substance being examined in about 5 ml of water. Add 2 ml of lead acetate solution, a white precipitate, soluble in ammonium acetate solution and in sodium hydroxide solution, is produced.

$$\underset{\substack{\text{Magnesium}\\ \text{sulphate}}}{MgSO_4} + \underset{\substack{\text{Lead}\\ \text{acetate}}}{(CH_3COO)_2\,Pb} \longrightarrow \underset{\substack{\text{Lead}\\ \text{sulphate}}}{PbSO_4} + \underset{\substack{\text{Sodium}\\ \text{acetate}}}{2CH_3COONa}$$

AMMONIUM CHLORIDE (NH_4Cl)

Category

Expectorant; diuretic, systemic acidifier.

Description

Colourless crystals or white crystalline powder.

Solubility

Freely soluble in water; sparingly soluble in ethanol.

Identification Tests

Ammonium chloride gives reactions for ammonium ions and chlorides.

Reactions of Ammonium Ions

1. Heat a few mg of the substance being examined with sodium hydroxide solution; ammonia is evolved, which is recognisable by its odour and by its action on moist red litmus paper, which turns blue.

$$\underset{\substack{\text{Ammonium}\\\text{chloride}}}{NH_4Cl} + \underset{\substack{\text{Sodium}\\\text{hydroxide}}}{NaOH} \longrightarrow \underset{\substack{\text{Sodium}\\\text{chloride}}}{NaCl} + \underset{\text{Water}}{H_2O} + \underset{\text{Ammonia}}{NH_3\uparrow}$$

2. Dissolve 10 mg of substance being examined in water. Add 1 ml of freshly prepared 10% w/v solution of sodium cobaltinitrite. A yellow precipitate is produced.

$$\underset{\substack{\text{Ammonium}\\\text{chloride}}}{3NH_4Cl} + \underset{\substack{\text{Sodium}\\\text{cobaltinitrite}}}{Na_3[Co(NO_2)_6]} \longrightarrow \underset{\substack{\text{Ammonium cobaltinitrite}\\\text{(yellow ppt)}}}{(NH_4)_3\,[Co(NO_2)_6]\downarrow} + \underset{\substack{\text{Sodium}\\\text{chloride}}}{3NaCl}$$

Reactions of Chlorides

1. Dissolve about 2 mg of sample in 2 ml of water. Acidify with dilute nitric acid and add 0.5 ml of silver nitrate solution. Shake and allow to stand. A curdy, white precipitate is formed, which is insoluble in nitric acid but soluble after being well washed with water in dilute ammonia solution, from which it is reprecipitated by the addition of dilute nitric acid.

$$\underset{\substack{\text{Sodium}\\\text{chloride}}}{NaCl} + \underset{\substack{\text{Silver}\\\text{nitrate}}}{AgNO_3} \longrightarrow \underset{\substack{\text{Silver}\\\text{chloride}\\\text{(white ppt)}}}{AgCl\downarrow} + \underset{\substack{\text{Sodium}\\\text{nitrate}}}{NaNO_3}$$

$$\underset{\substack{\text{Silver}\\\text{nitrate}}}{AgCl} + \underset{\substack{\text{Ammonium}\\\text{hydroxide}}}{2NH_4OH} \longrightarrow \underset{\text{Soluble}}{[Ag(NH_3)_2]Cl} + 2H_2O$$

2. Mix 10 mg of sample with 0.2 g of potassium dichromate in a test tube. Add 1 ml of sulphuric acid. Place a filter paper strip moistened with 0.1 ml of diphenylcarbazide solution, over the mouth of the test tube. The paper turns violet-red.

$$\underset{\substack{\text{Sodium}\\\text{chloride}}}{4NaCl} + \underset{\substack{\text{Potassium}\\\text{dichromate}}}{K_2Cr_2O_7} + \underset{\substack{\text{Sulphuric}\\\text{acid}}}{3H_2SO_4} \longrightarrow \underset{\substack{\text{Chromyl chloride}\\\text{(deep red)}}}{2CrO_2Cl_2\uparrow} + \underset{\substack{\text{Ammonium}\\\text{sulphate}}}{2(NH_4)_2SO_4} + \underset{\substack{\text{Potassium}\\\text{sulphate}}}{K_2SO_4} + 3H_2O$$

The deep red chromyl chloride vapours are evolved which react with diphenyl carbazide to give violet-red colour.

Experiment 7: Identification of Ammonium Cation and Chloride Anion in Ammonium Chloride

Date________________

AIM

To perform the identification tests for the given sample of ammonium chloride.

CHEMICALS REQUIRED

Ammonium chloride, sodium hydroxide, red litmus paper, sodium cobaltinitrite, nitric acid, silver nitrate solution, dilute ammonia solution, potassium dichromate, sulphuric acid, diphenyl carbazide solution.

APPARATUS REQUIRED

Test tubes, test tube stand, test tube holder, glass rod.

PROCEDURE

The sample gives the reactions for ammonium ions and chlorides.

REACTIONS OF AMMONIUM IONS

S. no.	*Test*	*Observation*	*Inference*
1.	Take a few mg of sample and add sodium hydroxide solution. Heat. Bring moist red litmus paper near the mouth of the test tube.		
2.	Dissolve 10 mg of sample in water. Add 1 ml of freshly prepared 10% w/v solution of sodium cobaltinitrite.		

REACTIONS OF CHLORIDES

S. no.	*Test*	*Observation*	*Inference*
1.	Dissolve 2 mg of sample in 2 ml of water. Acidify with dilute nitric acid and add 0.5 ml of silver nitrate solution. Shake and allow to stand. • Add dilute ammonia solution. • Add nitric acid.		
2.	Mix 10 mg of sample with 0.2 g of potassium dichromate in a test tube. Add 1 ml of sulphuric acid. Place a filter paper strip, moistened with 0.1 ml of diphenyl carbazide solution over the mouth of the test tube.		

RESULT

The sample gives the identification tests for ammonium chloride.

Experiment 8: Identification of Sodium Cation and Bicarbonate Anion in Sodium Bicarbonate

Date________________

AIM

To perform the identification tests for the given sample of sodium bicarbonate.

CHEMICALS REQUIRED

Sodium bicarbonate, phenolphthalein solution, potassium carbonate, potassium antimonate, acetic acid, magnesium uranyl acetate solution, magnesium sulphate solution, acetic acid, barium hydroxide, hydrochloric acid.

APPARATUS REQUIRED

Test tubes, test tube stand, test tube holder, glass rod.

PROCEDURE

S. no.	*Test*	*Observation*	*Inference*
1.	To the test solution add 0.1 ml of phenolphthalein solution. Heat		

The sample gives the reactions of sodium ions and bicarbonate ions.

REACTIONS OF SODIUM IONS

Prepare a 20% w/v solution of sodium chloride in carbon dioxide free distilled water (test solution).

S. no.	*Test*	*Observation*	*Inference*
1.	To 2 ml of test solution, add 2 ml of 15% w/v solution of potassium carbonate. Heat to boiling. Add 4 ml of potassium antimonate solution. Heat to boiling. Cool in ice and if necessary, scratch the inside of the test tube with a glass rod.		
2.	To 2 ml of test solution add 0.5 ml of 1 M acetic acid followed by large excess of magnessium uranyl acetate solution.		

Reactions of Bicarbonates

1. Boil the aqueous solution of the substance being examined. Carbon dioxide gas is liberated.

$$\underset{\substack{\text{Sodium}\\ \text{bicarbonate}}}{2NaHCO_3} \longrightarrow \underset{\substack{\text{Sodium}\\ \text{carbonate}}}{Na_2CO_3} + H_2O + CO_2\uparrow$$

2. Treat a solution of the substance being examined with a solution of magnesium sulphate; no precipitate is formed (distinction from carbonates). Boil; a white precipitate is formed.

$$\underset{\substack{\text{Sodium}\\ \text{bicarbonate}}}{2NaHCO_3} + \underset{\substack{\text{Magnesium}\\ \text{sulphate}}}{MgSO_4} \longrightarrow \underset{\substack{\text{Magnesium}\\ \text{carbonate}}}{MgCO_3\downarrow} + H_2O + CO_2 + \underset{\substack{\text{Sodium}\\ \text{sulphate}}}{Na_2SO_4}$$

3 . Dissolve 0.1 g of the substance being examined in 2 ml of water. Add 2 ml of 2 M acetic acid, close the tube immediately using a stopper fitted with a glass tube bent at two right angles. Heat gently and collect the gas in 5 ml of barium hydroxide solution. A white precipitate forms that dissolves on addition of an excess of dilute hydrochloric acid.

$$\underset{\substack{\text{Sodium}\\ \text{bicarbonate}}}{NaHCO_3} + \underset{\substack{\text{Acetic}\\ \text{acid}}}{CH_3COOH} \longrightarrow \underset{\substack{\text{Sodium}\\ \text{acetate}}}{CH_3COONa} + H_2O + CO_2\uparrow$$

$$\underset{\substack{\text{Carbon}\\ \text{dioxide}}}{CO_2\uparrow} + \underset{\substack{\text{Barium}\\ \text{hydroxide}}}{Ba(OH)_2} \longrightarrow \underset{\substack{\text{Barium}\\ \text{carbonate}}}{BaCO_3\downarrow} + H_2O$$

$$\underset{\substack{\text{Barium}\\ \text{carbonate}}}{BaCO_3} + \underset{\substack{\text{Hydrochloric}\\ \text{acid}}}{2HCl} \longrightarrow \underset{\substack{\text{Barium}\\ \text{chloride}}}{BaCl_2} + CO_2 + H_2O$$

POTASSIUM IODIDE (KI)

Category

Antithyroid; antifungal; expectorant.

Description

Colourless crystals or white powder, odourless.

Solubility

Very soluble in water; freely soluble in glycerin; soluble in ethanol.

Identification Tests

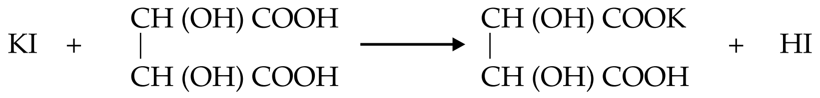
A 10% w/v solution in carbon dioxide free water (test solution) gives the reactions of potassium salts and of iodides.

Reactions of Potassium Ions

1. To 1 ml of test solution, add 1 ml of dilute acetic acid and 1 ml of freshly prepared 10% w/v solution of sodium cobaltinitrite; a yellow or orange-yellow precipitate is produced immediately.

$$\underset{\substack{\text{Potassium}\\\text{iodide}}}{3KI} + \underset{\substack{\text{Sodium}\\\text{cobaltinitrite}}}{Na_3\,[Co\,(NO_2)_6]} \longrightarrow \underset{\substack{\text{Potassium}\\\text{cobaltinitrite}\\\text{(yellow ppt)}}}{K_3\,[Co(NO_2)_6]} + \underset{\substack{\text{Sodium}\\\text{iodide}}}{3NaI}$$

2. To 2 ml of test solution, add 1 ml of sodium carbonate solution and heat; no precipitate is formed. Add 0.05 ml of sodium sulphide solution; no precipitate is formed. Cool in ice, add 2 ml of a 15% w/v solution of tartaric acid and allow to stand; a white, crystalline precipitate is produced.



$$\underset{\substack{\text{Potassium}\\\text{iodide}}}{KI} + \underset{\text{Tartaric acid}}{\begin{array}{l}CH\,(OH)\,COOH\\|\\CH\,(OH)\,COOH\end{array}} \longrightarrow \underset{\substack{\text{Potassium}\\\text{hydrogen tartrate}\\\text{(white ppt)}}}{\begin{array}{l}CH\,(OH)\,COOK\\|\\CH\,(OH)\,COOH\end{array}} + \underset{\substack{\text{Hydroiodic}\\\text{acid}}}{HI}$$

3. Ignite a few mg of substance being examined, cool and dissolve in the minimum quantity of water. To this solution, add 1 ml of platinic chloride (hexachloro platinic acid) solution in the presence of 1 ml of hydrochloric acid; a yellow, crystalline precipitate is produced.

$$\underset{\substack{\text{Potassium}\\\text{iodide}}}{2KI} + \underset{\substack{\text{Hexachloro-}\\\text{platinic acid}}}{H_2[PtCl_6]} \longrightarrow \underset{\substack{\text{Potassium}\\\text{hexachloroplatinate}\\\text{(yellow ppt)}}}{K_2[PtCl_6]\downarrow} + \underset{\substack{\text{Hydroiodic}\\\text{acid}}}{2HI}$$

Reactions of Iodides

1. Take 2 ml of test solution; acidify with and dilute nitric acid and add 0.5 ml of silver nitrate solution. Shake and allow to stand; a curdy, pale yellow precipitate is formed.

$$\underset{\substack{\text{Potassium}\\\text{iodide}}}{KI} + \underset{\substack{\text{Silver}\\\text{nitrate}}}{AgNO_3} \xrightarrow{HNO_3} \underset{\substack{\text{Silver}\\\text{iodide}\\\text{(yellow ppt)}}}{AgI} + \underset{\substack{\text{Potassium}\\\text{nitrate}}}{KNO_3}$$

Filter and wash the precipitate with water. Suspend the precipitate in 2 ml of water and add 1.5 ml of ammonia; the precipitate does not dissolve.

2. To 0.2 ml of test solution, add 0.5 ml of 1 M sulphuric acid, 0.15 ml of potassium dichromate solution, 2 ml of water and 2 ml of chloroform, shake for a few seconds and allow to stand; the chloroform layer is violet or violet-red. Potassium iodide is oxidised by potassium dichromate and liberates iodine. This iodine dissolves in chloroform and imparts violet colour.
3. To 1 ml of test solution add 0.5 ml of mercuric chloride solution; a dark red precipitate is formed which is slightly soluble in an excess of this reagent and very soluble in an excess of potassium iodide solution due to formation of potassium mercury iodide.

$$\underset{\substack{\text{Potassium}\\\text{iodide}}}{2KI} + \underset{\substack{\text{Mercuric}\\\text{chloride}}}{HgCl_2} \longrightarrow \underset{\substack{\text{Mercuric}\\\text{iodide}\\\text{(red ppt)}}}{HgI_2} + \underset{\substack{\text{Potassium}\\\text{chloride}}}{2KCl}$$

BARIUM SULPHATE ($BaSO_4$)

Category

Diagnostic aid (radiopaque medium for gastrointestinal tract.)

Description

Fine, heavy, white powder, free from gritty particles, odourless.

Solubility

Practically insoluble in water, in organic solvents and in dilute solutions of acids and of alkalis.

Identification Tests

1. Boil 0.2 g with 5 ml of a 50% w/v solution of sodium carbonate for 5 minutes, add 10 ml of water and filter. Reserve the residue for test of barium ions. Acidify the filterate with dilute hydrochloric acid. Barium sulphate reacts with sodium carbonate to give less soluble barium carbonate and soluble sodium sulphate.

$$\underset{\substack{\text{Barium}\\ \text{sulphate}}}{BaSO_4} + \underset{\substack{\text{Sodium}\\ \text{carbonate}}}{Na_2CO_3} \longrightarrow \underset{\substack{\text{Barium}\\ \text{carbonate}}}{BaCO_3} + \underset{\substack{\text{Sodium}\\ \text{sulphate}}}{Na_2SO_4}$$

The test for barium is carried out on residue and filtrate is tested for sulphate ions.

Tests of Sulphate Ions

1. Take 1 ml of the filtrate, acidify with dilute hydrochloric acid and add 0.2 ml of barium chloride solution. A white precipitate of barium sulphate is formed.

$$\underset{\substack{\text{Sodium}\\ \text{sulphate}}}{Na_2SO_4} + \underset{\substack{\text{Barium}\\ \text{chloride}}}{BaCl_2} \longrightarrow \underset{\substack{\text{Barium}\\ \text{sulphate}\\ \text{(white ppt)}}}{BaSO_4\downarrow} + \underset{\substack{\text{Sodium}\\ \text{chloride}}}{2NaCl}$$

2. Add 0.1 ml of iodine solution to the suspension obtained in the above test, the suspension remains yellow but is decolourised by adding, dropwise, stannous chloride solution. Boil the mixture. No coloured precipitate appears.
3. To 1 ml of the filtrate, add 0.4 ml of lead acetate solution. A white precipitate is obtained.

$$\underset{\substack{\text{Sodium}\\ \text{sulphate}}}{Na_2SO_4} + \underset{\substack{\text{Lead}\\ \text{acetate}}}{(CH_3COO)_2Pb} \longrightarrow \underset{\substack{\text{Lead}\\ \text{sulphate}\\ \text{(white ppt)}}}{PbSO_4\downarrow} + \underset{\substack{\text{Sodium}\\ \text{acetate}}}{2CH_3COONa}$$

Divide the suspension in two portions.

1. To one portion, add ammonium acetate solution, the precipitate dissolves.
2. To another portion, add sodium hydroxide solution. The precipitate dissolves.

Experiment 10: Identification of Barium Cation and Sulphate Anion in Barium Sulphate

Date________________

AIM

To perform the identification tests for the given sample of barium sulphate.

CHEMICALS REQUIRED

Barium sulphate, sodium carbonate, dilute hydrochloric acid, barium chloride solution, iodine solution, stannous chloride solution, lead acetate solution, ammonium acetate solution, sodium hydroxide solution, sulphuric acid.

APPARATUS REQUIRED

Test tubes, test tube stand, test tube holder, glass rod.

PROCEDURE

Boil 0.2 g of sample with 5 ml of 50% w/v solution of sodium carbonate for 5 minutes, add 10 ml of water and filter. Use the filtrate for tests of sulphates while residue for tests of barium.

TESTS OF SULPHATE IONS

S. no.	*Test*	*Observation*	*Inference*
1.	Take 1 ml of filtrate, acidify with dilute hydrochloric acid and add 0 .2 ml of barium chloride solution.		
2.	Add 0.1 ml of iodine solution to the suspension obtained in above test. Add stannous chloride solution dropwise. Boil the mixture.		
3.	Take 1 ml of the filtrate, add 0.4 ml of lead acetate solution. Divide the suspension in two portions. a. Add ammonium acetate solution to first portion. b. Add sodium hydroxide solution to second portion.		

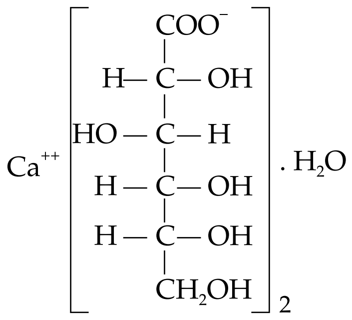
TESTS OF BARIUM IONS

S. no.	*Test*	*Observation*	*Inference*
1.	Wash the residue three times with sucessive small quantities of water. Add 5 ml of dilute hydrochloric acid to the residue, filter • Add to the filtrate dilute sulphuric acid. • Add sodium hydroxide solution.		

RESULT

The given sample gives identification tests for barium sulphate.

CALCIUM GLUCONATE



Calcium gluconate is calcium D-gluconate monohydrate.

Category

Calcium replenisher.

Description

White, crystalline powder or granules.

Solubility

Sparingly soluble in water but freely soluble in boiling water; insoluble in ethanol.

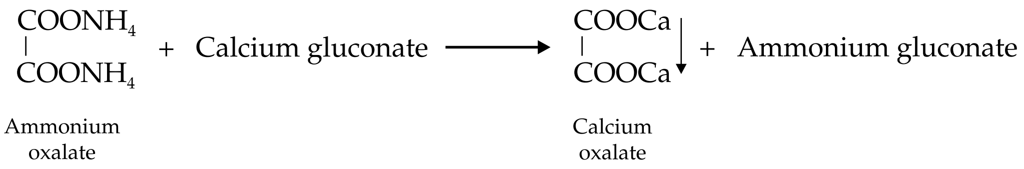
Identification Tests

Tests of Calcium Salts

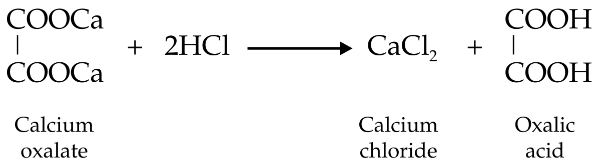
1. Dissolve 20 mg of the substance being examined in 5 ml of 5 M acetic acid. Add 0.5 ml of potassium ferrocyanide solution; the solution remains clear. Add about 50 mg of ammonium chloride, a white, crystalline precipitate is formed.

$$\underset{\text{gluconate}}{\text{Calcium}} + \underset{\substack{\text{Potassium}\\ \text{ferrocyanide}}}{K_4[Fe(CN)_6]} + \underset{\substack{\text{Ammonium}\\ \text{chloride}}}{2NH_4Cl} \longrightarrow \underset{\substack{\text{Ammonium calcium}\\ \text{ferrocyanide}}}{(NH_4)_2Ca\,[Fe(CN)_6]} + 2KCl + \underset{\text{gluconate}}{\text{Potassium}}$$

2. To 5 ml of a 0.4 % w/v solution of the substance being examined, add 0.2 ml of a 2% w/v solution of ammonium oxalate. A white precipitate that is only sparingly soluble in dilute acetic acid but soluble in hydrochloric acid is obtained.

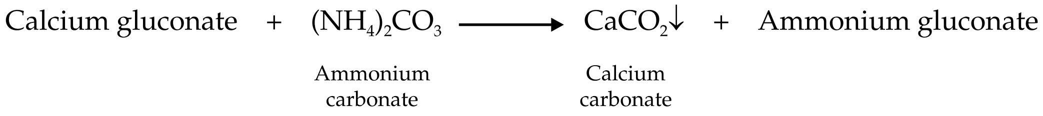


$$\underset{\substack{\text{Ammonium}\\ \text{oxalate}}}{\begin{array}{c}COONH_4\\ |\\ COONH_4\end{array}} + \text{Calcium gluconate} \longrightarrow \underset{\substack{\text{Calcium}\\ \text{oxalate}}}{\begin{array}{c}COOCa\\ |\\ COOCa\end{array}}\downarrow + \text{Ammonium gluconate}$$



$$\underset{\substack{\text{Calcium}\\ \text{oxalate}}}{\begin{array}{c}COOCa\\ |\\ COOCa\end{array}} + 2HCl \longrightarrow \underset{\substack{\text{Calcium}\\ \text{chloride}}}{CaCl_2} + \underset{\substack{\text{Oxalic}\\ \text{acid}}}{\begin{array}{c}COOH\\ |\\ COOH\end{array}}$$

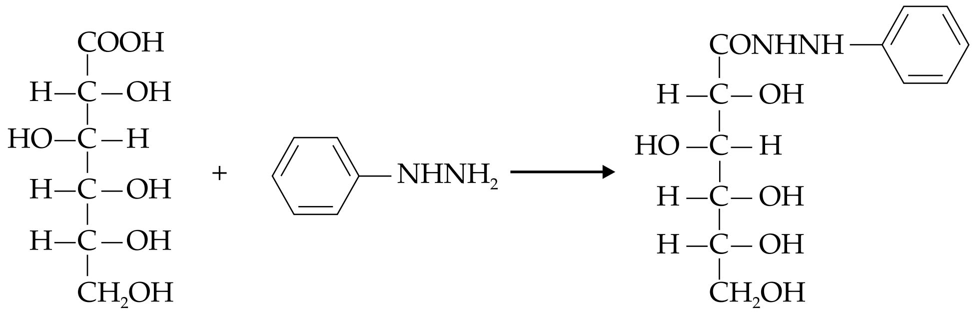
3. Dissolve 20 mg of the substance being examined in minimum quantity of dilute HCl and neutralise with dilute sodium hydroxide solution. Add 5 ml of ammonium carbonate solution; a white precipitate is formed which after boiling and cooling the mixture, is only sparingly soluble in ammonium chloride solution.



$$\text{Calcium gluconate} + \underset{\text{Ammonium carbonate}}{(NH_4)_2CO_3} \longrightarrow \underset{\text{Calcium carbonate}}{CaCO_2\downarrow} + \text{Ammonium gluconate}$$

Reactions of Gluconic Acid

1. To 1 ml of a 3% w/v solution, add 0.05 ml of ferric chloride test solution; a yellow colour is produced due to formation of ferric gluconate.
2. To 0. 75 gm of sample in 7.5 ml of warm water, add 1 ml of glacial acetic acid and 1.5 ml of freshly distilled phenylhydrazine. Heat the mixture on waterbath for 30 minutes. Allow to cool. Scratch the inner surface of the tube with a glass rod, until crystals of gluconic acid phenyl hydrazide begin to form. Set aside the mixture for 10 minutes. Filter, dissolve the precipitate in 10 ml of hot water and add a small amount of decolourising charcoal. Filter the suspension. Allow the filtrate to cool and scratch the inner surface of the tube. White crystals are obtained. Dry them and take the melting point. Observe that the product melts at 200°, with decomposition.



Gluconic acid Phenyl hydrazine Gluconic acid phenyl hydrazide

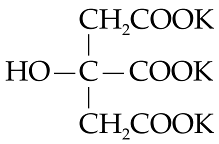
REACTIONS OF GLUCONIC ACID

S. no.	*Test*	*Observation*	*Inference*
1.	Take 1 ml of 3% w/v solution of sample and add 0.05 ml of ferric chloride test solution.		
2.	To 0.75 gm of sample in 7.5 ml of warm water, add 1 ml of glacial acetic acid and 1.5 ml of freshly distilled phenyl hydrazine. Heat the mixture on water bath for 30 minutes. Allow to cool. Scratch the inner surface of the tube with a glass rod. Filter the precipitate and dissolve it in 10 ml of hot water. Add small amount of decolourising charcoal. Filter the suspension. Allow the filtrate to cool. Scratch the inner surface of the tube. Dry the crystals. Take the melting point.		

RESULT

The sample gives identification tests for calcium gluconate.

POTASSIUM CITRATE



$$\begin{array}{c} \qquad\ \ CH_2COOK \\ \qquad | \\ HO-C-COOK \\ \qquad | \\ \qquad\ \ CH_2COOK \end{array}$$

Category

Systemic alkaliniser.

Description

White, granular crystals or crystalline powder; odourless; hydroscopic.

Solubility

Very soluble in water; soluble in glycerin; practically insoluble in ethanol.

Identification Tests

Potassium citrate gives reactions for potassium and citrate ions.

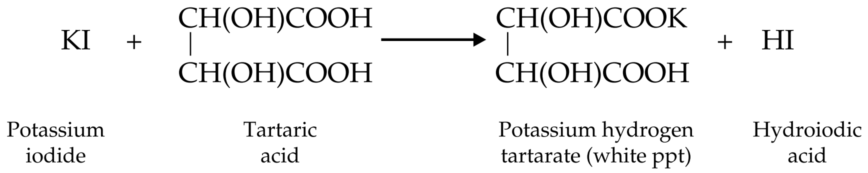
Test solution for reactions of potassium salts: Prepare 10% w/v solution in carbon dioxide free distilled water.

Reactions of Potassium Ions

1. To 1 ml of test solution, add 1 ml of dilute acetic acid and 1 ml of freshly prepared 10% w/v solution of sodium cobaltinitrite; a yellow or orange-yellow precipitate is produced immediately.

$$\underset{\text{Potassium iodide}}{3KI} + \underset{\text{Sodium cobaltinitrite}}{Na_3[Co(NO_2)_6]} \longrightarrow \underset{\text{Potassium cobaltinitrite}}{K_3[Co(NO_2)_6]} + \underset{\text{Sodium iodide}}{3NaI}$$

2. To 2 ml of test solution, add 1 ml of sodium carbonate solution and heat; no precipitate is formed. Add 0.05 ml of sodium sulphide solution; no precipitate is formed. Cool in ice, add 2 ml of a 15% w/v solution of tartaric acid and allow to stand; a white, crystalline precipitate is produced.



$$\underset{\text{Potassium iodide}}{KI} + \underset{\text{Tartaric acid}}{\begin{array}{l} CH(OH)COOH \\ | \\ CH(OH)COOH \end{array}} \longrightarrow \underset{\text{Potassium hydrogen tartarate (white ppt)}}{\begin{array}{l} CH(OH)COOK \\ | \\ CH(OH)COOH \end{array}} + \underset{\text{Hydroiodic acid}}{HI}$$

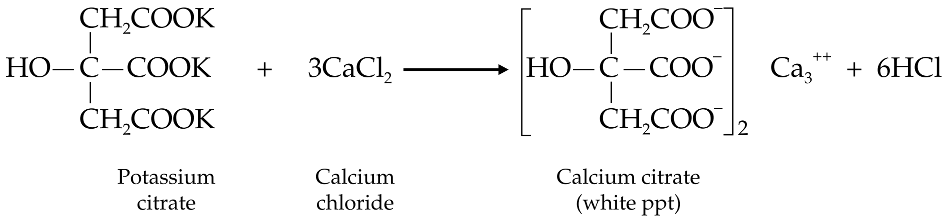
3. Ignite a few mg of substance being examined, cool and dissolve in the minimum quantity of water. To this solution, add 1 ml of platinic chloride (hexachloroplatinic acid) solution in the presence of 1 ml of hydrochloric acid; a yellow, crystalline precipitate is produced.

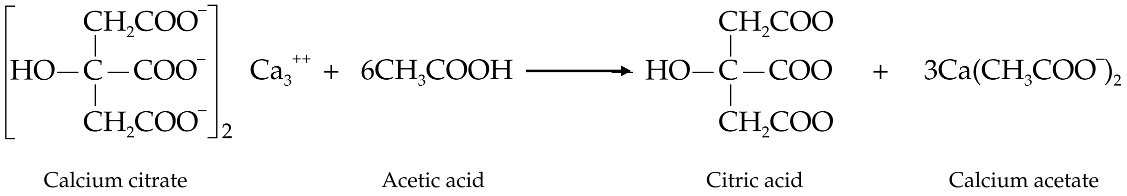
$$\underset{\text{Potassium iodide}}{2KI} + \underset{\text{Hexachloroplatinic acid}}{H_2[PtCl_6]} \longrightarrow \underset{\text{Potassium hexachloroplatinate (yellow ppt)}}{K_2[PtCl_6]\downarrow} + \underset{\text{Hydroiodic acid}}{2HI}$$

Reactions of Citrate Ions

Test solution: Prepare 2% w/v solution in carbon dioxide free distilled water.

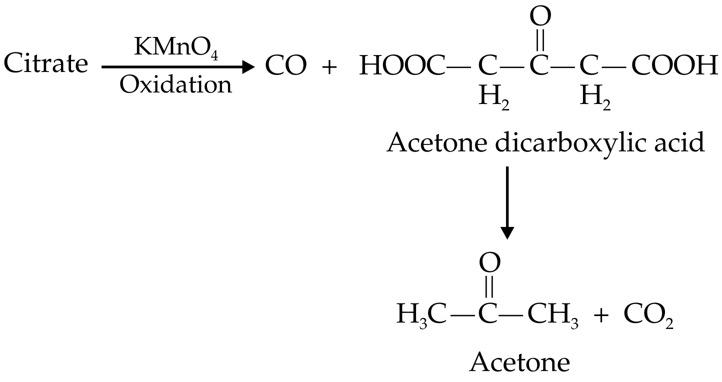
1. To a neutral test solution add a solution of calcium chloride. No precipitate is produced. Boil the solution. A white precipitate of calcium citrate, soluble in 6 M acetic acid is produced.





2. Take 5 ml of the test solution, add 0 .5 ml of sulphuric acid and 3 ml of potassium permanganate solution. Warm until colour of the permanganate is discharged. Add 0.5 ml of a 10% w/v solution of sodium nitroprusside in 1 M sulphuric acid and 4 g of sulphamic acid (H_2NSO_3H). Make alkaline with strong ammonia solution, added dropwise until all the sulphamic acid has dissolved. On addition of an excess of strong ammonia solution a violet colour, which turns to violet-blue, is produced.

   Citrate ions decolourise acidified potassium permanganate due to oxidation into carbon monoxide and acetone dicarboxylic acid which further undergoes conversion to acetone and carbon dioxide.



Acetone dicarboxylic acid reacts with sodium nitroprusside, $Na_2[Fe(CN)_5NO]2H_2O$, in presence of sulphamic acid to give violet colour which changes to violet-blue.

REACTIONS OF CITRATE IONS

S. no.	Test	Observation	Inference
1.	To a neutral test solution, add a solution of calcium chloride. Boil the		
2.	To 5 ml of test solution, add 0.5 ml of sulphuric acid and 3 ml of potassium permanganate solution. Warm until colour of the permanganate is discharged. Add 0.5 ml of 10% w/v solution of sodium nitroprusside in 1 M sulphuric acid and 4 g of sulphamic acid. Add dropwise strong ammonia solution until all sulphamic acid has dissolved. Add excess of strong ammonia solution.		

RESULT

The sample gives the identification tests for potassium citrate.