

BGS SCIENCE ACADEMY & RESEARCH CENTER
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IV Semester B.Sc.,
Chemistry Laboratory Manual



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Containing two acid and two basic radicals.(10 experiments)**
- 2. Separation of Fe^{2+} and Mg^{2+} from a mixture by Solvent Extraction method.**
- 3. Estimation of C.O.D. in the given sample of effluent.**

**SYSTEMATIC PROCEDURE FOR SEMI-MICRO QUALITATIVE ANALYSIS
OF INORGANIC SALT MIXTURES CONTAINING TWO SIMPLE SALTS**

The scheme of analysis of salt mixture involves mainly the following steps

1. Preliminary tests, 2. Detection of acid radicals 3. Detection of basic radicals 4. Systematic report

1. Preliminary tests:

- a) Physical state: Solid
- b) Appearance: Crystalline/amorphous
- c) Color:
- d) Solubility: The solubility to be tested with various solvents first with cold and then with hot condition in the following order of solvents: i) water, ii) dil.HCl iii) dil.HNO₃ iv) con.HNO₃

2. Detection of acid radicals: The acid radicals are classified into three groups given below based on reaction with the group reagent

Group No.	Group reagent	Radicals
I	Dil. HCl acid	Carbonate(CO ₃ ²⁻), Bicarbonate(HCO ₃ ⁻)
II	Con. H ₂ SO ₄	Chloride(Cl ⁻), Bromide(Br ⁻) Nitrate(NO ₃ ⁻)
III	-----	Borate(BO ₃ ³⁻), Phosphate(PO ₄ ³⁻) Sulphate(SO ₄ ²⁻)

Detection of I group acid radicals:

Experiment	Observation	Inference
Salt mixture+ dil.Hydrochloric acid in a semi-micro test tube, Nature of the evolved gas is observed.	Brisk effervescence It is colorless odour less gas	I group acid radical is Present May be carbonate or bicarbonate

<p><u>Test for carbonate/bicarbonate</u> Salt mixture + dil.HCl, the evolved gas is passed through a test tube containing lime water</p> <p><u>Confirmatory tests:</u> Salt mixture+ water, the contents of the test tube is boiled, the evolved gas is passed through a test tube containing lime water</p>	<p>Lime water turns milky</p> <p>i) Lime water turns milky</p> <p>ii) Lime water does not turn milky</p>	<p>Carbonate or bicarbonate is present</p> <p>Bi-carbonate is confirmed</p> <p>Carbonate is Confirmed</p>
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Detection of II Group Acid radicals

<p>Salt mixture is taken in a dried test tube + Con. Sulphuric acid (5 drops) If no reaction takes place in cold, the test tube is heated, Nature of the evolved gas is observed</p> <p><u>Tests for Chloride(Cl⁻)</u> Salt mixture + Con. Sulphuric- acid, a glass rod dipped in ammonium hydroxide solution is exposed to the outgoing gas.</p> <p><u>Chromyl chloride test</u> (Confirmatory Test for chloride) Salt mixture+ potassium dichromate crystals are taken in a dry test tube + Con.H₂SO₄, the contents are heated, the red vapours evolved is passed into a test tube containing water + NH₄OH + acetic acid + lead acetate, shaken well.</p>	<p>Vigorous reaction</p> <p>i) Colorless fuming gas ii) Reddish brown fumes in cold iii) Reddish brown fumes on heating</p> <p>Dense white fumes</p> <p>Bright yellow precipitate</p>	<p>II group acid radicals are present</p> <p>May be chloride May be bromide May be nitrate</p> <p>Chloride is present</p> <p>Chloride is confirmed</p>
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<p><u>Test for Bromide(Br⁻):</u> Salt mixture+Con.sulphuric acid</p> <p><u>Confirmatory test for Bromide (Globule test):</u> Salt solution + Carbon tetrachloride + 10 drops of chlorine water, shaken well</p>	<p>Reddish brown fumes</p> <p>Orange red globule</p>	<p>Bromide is present</p> <p>Bromide is confirmed</p>
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<p><u>Test for Nitrate(NO₃⁻)</u> Salt mixture + copper turnings + Con.sulphuric acid, the contents are heated.</p>	<p>Intense reddish brown fumes</p>	<p>Nitrate is confirmed</p>
<p><u>Confirmatory test for Nitrate</u> <u>(Brown ring test):</u> Salt solution + Freshly prepared ferrous sulphate soln.(if precipitate appears,centrifuged. To the filtrate, con.sulphuric acid is added along the sides of the test tube slowly.</p>	<p>Brown ring is formed at the junction of the two solutions</p>	<p>Nitrate is confirmed</p>

Detection of III Group Acid radicals

<p><u>Test for Borate(BO₃³⁻)</u> Salt mixture+ 10 drops of con. Sulphuric acid + ethyl alcohol heated, the vapours coming out of the test tube are ignited</p>	<p>Vapours burns with Green edged flame</p>	<p>Borate is confirmed</p>
<p><u>Test for Phosphate(PO₄³⁻)</u> Salt solution + con. Nitric acid heated, cooled + 1ml of ammonium molybdate solution</p>	<p>Bright yellow precipitate</p>	<p>Phosphate is Confirmed</p>
<p><u>Test for Sulphate(SO₄²⁻)</u> Salt solution+dil.HCl acid + Barium chloride solution. To the above mixture excess of dil. hydrochloric acid is added.</p>	<p>White precipitate White precipitate is insoluble</p>	<p>Sulphate is present Sulphate is confirmed</p>

III. DETECTION OF BASIC RADICALS

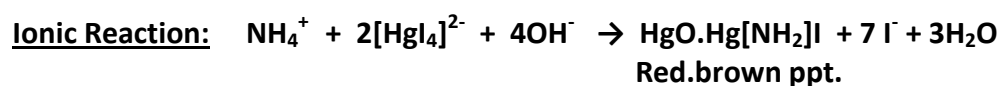
Preparation of original solution : About 5-10mg of the salt mixture treated with the following solvents first in the cold condition and later in hot condition. 1.Water 2. Dil.HCl 3) Dil.HNO₃ . About quarter a test tube of the solvent + salt mixture in small portions with shaking to get a saturated solution(O.S). The following table gives the classification of the basic radicals into various groups based on its reaction with the group reagent.

Group No.	Group Reagent	Radicals	Composition of the precipitate	Color of the Precipitate
I	Dil.HCl	Pb ²⁺	PbCl ₂	White
II	Dil.HCl + H ₂ S*	Bi ³⁺ Cu ²⁺ Cd ²⁺	Bi ₂ S ₃ CuS CdS	Brown Black Yellow
III	NH ₄ Cl(s) + NH ₄ OH(excess)	Fe ²⁺ Fe ³⁺ Al ³⁺	Fe(OH) ₂ Fe(OH) ₃ Al(OH) ₃	Green Red. Brown Gel.white
IV	NH ₄ Cl(s) + NH ₄ OH(ex) + H ₂ S*	Zn ²⁺ Mn ²⁺	ZnS MnS	White Buff
V	NH ₄ Cl(s) + NH ₄ OH(ex) + (NH ₄) ₂ CO ₃	Ba ²⁺ Sr ²⁺ Ca ²⁺	Ba CO ₃ Sr CO ₃ Ca CO ₃	White White White
	*Thioacetamide solution in water can be used instead of hydrogen sulphide	NH ₄ ⁺ Mg ²⁺ Na ⁺ K ⁺	-- -- -- --	-- -- -- --

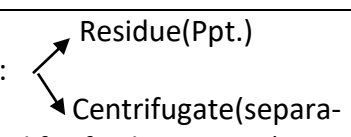
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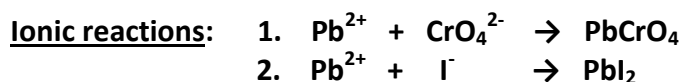
- About 1ml of the original solution is used for each group tests.
- If precipitate is found in any group test of basic radicals, the precipitation must be done by adding reagent of that group to the remaining part of the original solution for complete precipitation and centrifuged. The above precipitate is to be used for that group and the clear centrifugate is used for subsequent group analysis.
- If the previous groups are absent then use directly the O.S. or salt mixture.
- Since the test for ammonium radical is carried out directly with the salt mixture and also the presence of other metal ions does not interfere with its test, it is carried out in the beginning of the analysis of basic radicals
- Liquor ammonia can be added slowly instead of ammonium hydroxide till the solution smells ammonia.
- For II and IV group analysis Thio-acetamide solution in water or sodium sulphide solution can be used instead of hydrogen sulphide.

Experiment	Observation	Inference
<p>TEST FOR AMMONIUM RADICAL(NH₄⁺) 5-10mg of the salt mixture + 1ml of NaOH solution, the contents of the test tube is heated A moist red litmus paper is exposed to the out coming gas</p> <p>Confirmatory test for NH₄⁺ Nessler's reagent test : 10mg of the salt mixture + 1ml of sodium hydroxide solution,boiled and the vapours are passed into a test tube containg Nessler's reagent</p>	<p>Pungent odoured gas is liberated</p> <p>Red litmus turns blue</p> <p>Reddish brown precipitate</p>	<p>VI Group basic radical is present May be Ammonium radical Ammonium radical is present(NH₄⁺)</p> <p>Ammonium is Cofirmed(NH₄⁺)</p>

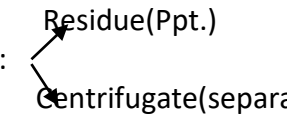


Detection of I Group Basic Radicals

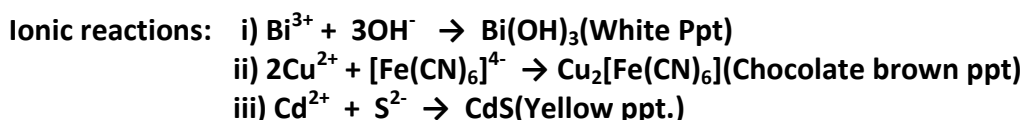
1 ml of the original solution + 1ml of dil HCl in excess	White precipitate	I group basic radical is present
<p>Centrifuge: </p> <p>Above group residue + 5ml of Water, boiled and cooled. Above hot solution is divided into two parts and tested as follows:</p> <p>Confirmatory tests for lead</p> <p>1. Potassium Chromate test: I part + acetic acid +Potassium Chromate solution.</p> <p>2. Golden yellow spangles test II part + 1ml of potassium iodide solution, the yellow precipitate obtained is dissolved in excess of water and boiled, the contents are cooled under the tap slowly.</p>	<p>Residue dissolves in hot condition and reappears on cooling</p> <p>Bright Yellow Precipitate</p> <p>Bright Yellow Precipitate</p> <p>Golden Yellow Spangles</p>	<p>Pb²⁺ is present</p> <p>Pb²⁺ is confirmed</p> <p>Pb²⁺ is confirmed</p>



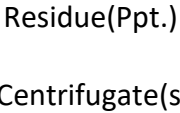
Detection of II Group Basic Radicals

<p>About 1ml of the above group centrifugate(or original solution if I group is absent) + dil HCl +H₂S gas is passed or thioacetamide solution(if no precipitate is observed, make sure by diluting with 2-3 drops of water). Color of the precipitate is observed</p> <p>Centrifuge:  </p> <p>Above group residue + 10 drops of yellow ammonium sulphide +NaOH solution, heated. Above step residue + 1ml of dil.Nitric acid, heated. Above step clear solution dil.H₂SO₄ + Ethanol, gently stirred.</p>	<p>Colored precipitate</p> <p>i) Black precipitate ii) Dark brown precipitate iii) Yellow precipitate (on dilution)</p> <p>Precipitate remains</p> <p>Precipitate dissolves</p> <p>No change</p>	<p>II group basic radical is present</p> <p>May be Cu²⁺ May be Bi³⁺ May be Cd²⁺</p> <p>II 'A' groups basic radical is present May be Bi³⁺, Cu²⁺ or Cd²⁺</p> <p>lead is absent</p>
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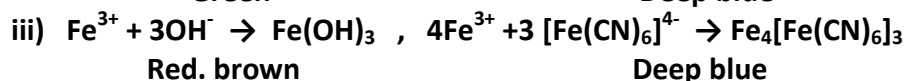
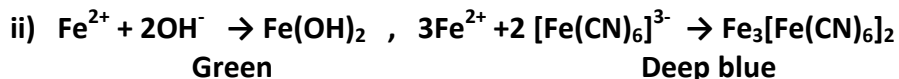
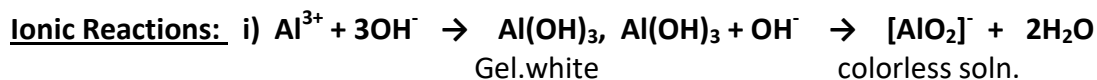
<p><u>Confirmatory test for Bi³⁺</u>: Above step residue + 4 drops of Con.HCl, clear solution + NaOH + Stannous chloride solution</p> <p><u>Confirmatory test for Cu²⁺</u>:(If the solution is blue then only this test is performed) Above step blue colored solution + acetic acid + potassium ferro-cyanide solution</p> <p><u>Confirmatory test for Cd²⁺</u> : Above step clear solution+3 drops of water + H₂S gas is passed or thioacetamide solution, warmed</p>	<p>Precipitate dissolves</p> <p>Black brown precipitate</p> <p>Chocolate brown precipitate</p> <p>Yellow precipitate</p>	<p>Bi³⁺ is confirmed</p> <p>Cu²⁺ is confirmed</p> <p>Cd²⁺ is confirmed</p>
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Detection of III Group Basic Radicals

<p>Centrifugate from II group is boiled off to expel off all H₂S, 2drops of con.HNO₃ or 1ml of salt solution if the first two groups are absent + solid NH₄Cl till saturated + excess of liquor ammonia till it smells sufficiently</p> <p>Color and nature of the precipitate is observed</p> <p>Centrifuge: </p>	<p>Precipitate is obtained</p> <p>i) Gelatinous white ii) Dirty green iii) Reddish brown</p> <p>i) Green or reddish brown precipitate ii) Clear solution</p>	<p>III Group Basic Radical is present May be Al³⁺ May be Fe²⁺ May be Fe³⁺</p> <p>i) May be Fe²⁺/ Fe³⁺ ii) Al³⁺ is present</p>
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<p><u>Confirmatory test for Al³⁺:</u> Above clear solution + Solid amm ammonium chloride, shaken well and boiled and cooled</p> <p><u>Confirmatory test for Fe²⁺:</u> Above step residue + 10 drops of Dil.HCl + 10 drops of Potassium Ferricyanide solution</p> <p><u>Confirmatory test for Fe³⁺:</u> Above step residue + 10 drops of Dil.HCl + 10 drops of Potassium ferrocyanide solution</p>	<p>Gelatinous white precipitate</p> <p>Deep blue precipitate</p> <p>Deep blue precipitate</p>	<p>Al³⁺ is confirmed</p> <p>Fe²⁺ is confirmed</p> <p>Fe³⁺ is confirmed</p>
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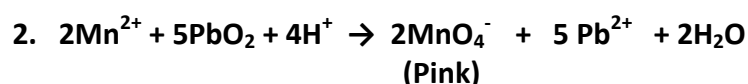


Detection of IV Group Basic Radicals

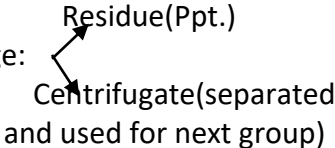
<p>Centrifugate from the above group(or original solution if the above groups are absent) + Solid NH₄Cl + Excess of liquor ammonia + H₂S gas is passed or thioacetamide solution and warmed Color of the precipitate is observed</p> <p>Centrifuge: </p> <p>Above group residue + a few drops of dil. HCl, heated</p>	<p>Precipitate is formed</p> <p>i) Dull white or white ii) Buff</p> <p>Precipitate dissolves</p>	<p>IV group Basic Radical is present</p> <p>May be zinc May be manganese</p> <p>May be Zn²⁺ or Mn²⁺</p>
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<p><u>Distinction be Zn²⁺ and Mn²⁺:</u> Above solution is boiled with 2 drops of con. nitric acid to expel off all H₂S + NaOH solution is added in dropwise and added in excess.</p> <p><u>Confirmatory test for Zn²⁺:</u> Above clear solution + 5 drops of acetic acid + 5 drops of potassium ferrocyanide solution</p> <p><u>Confirmatory test for Mn²⁺:</u> (Permanganic acid test): Above step flesh or buff precipitate + 5drops of water + a pinch of lead peroxide + few drops of con.nitric acid, boiled for 3 mins with stirring, diluted with water and allowed to stand for some time.</p>	<p>i) Clear solution ii) Buff or brown precipitate</p> <p>White precipitate</p> <p>Pink coloration in the supernatant portion of the liquid</p>	<p>Zn²⁺ is present Mn²⁺ is present</p> <p>Zn²⁺ is confirmed</p> <p>Mn²⁺ is confirmed</p>
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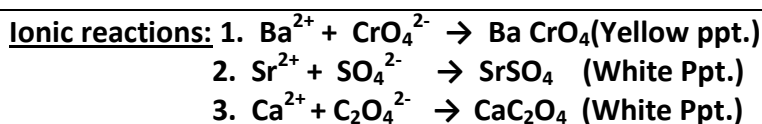
Ionic Reactions: 1. $Zn^{2+} + [Fe(CN)_6]^{4-} \rightarrow Zn_2[Fe(CN)_6]$ (White ppt.)



Detection of V Group Basic Radicals

<p>Centrifugate from IV group is boiled with 2 drops of con.nitric acid to expel off all H₂S(or Original solution if the above four groups are absent) + Solid NH₄Cl + excess of liquor ammonia + ammonium carbonate solution in excess.</p> <p>Centrifuge: </p>	<p>White precipitate</p>	<p>V group Basic Radical is present</p>
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<p>Above group residue is divided into two parts, one part of it is preserved to carry out flame test. Another part is dissolved in acetic acid and divided into 3 parts.</p> <p>Test for Ba²⁺: I part + potassium chromate solution. <u>Confirmatory test:- Flame test</u></p> <p>Above step residue or II part of the V group residue + a drop of Con. HCl, flame test is conducted</p> <p>Test for Sr²⁺: II part + Saturated ammonium sulphate solution. <u>Confirmatory test:- Flame test</u></p> <p>Above step residue or II part of the V group residue + a drop of Con. HCl, flame test is conducted</p> <p>Test for Ca²⁺: III part + ammo-nium oxalate solution, shaken well <u>Confirmatory test:- Flame test</u></p> <p>Above step residue or II part of the V group residue + a drop of Con. HCl, flame test is conducted</p>	<p>i) Yellow precipitate ii) Yellow solution</p> <p>Apple green colored flame White precipitate No Precipitate</p> <p>Crimson red colored flame</p> <p>White precipitate</p> <p>Brick red colored flame</p>	<p>Ba²⁺ is present Ba²⁺ is absent</p> <p>Ba²⁺ is confirmed</p> <p>Sr²⁺ is present Sr²⁺ is absent</p> <p>Sr²⁺ is confirmed</p> <p>Ca²⁺ is present</p> <p>Ca²⁺ is confirmed</p>
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VI Group Basic Radicals

V Group centrifugate or original solution (if ammonium present or previous groups are absent) is divided into two unequal parts and tested as follows.

<p>Test for Mg^{2+}: Smaller part+solid NH_4Cl + Excess of liquor NH_3 +10 drops of ammonium hydrogen Phosphate solution, shaken well.</p>	<p>White crystalline precipitate</p>	<p>Mg^{2+} is confirmed</p>
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Tests for Na^+ and K^+ : Larger part of the V group centrifugate or original solution+ 1-2 drops of con.HCl and the solution is evaporated to dryness in a small porcelain crucible with stirring till no more fumes are liberated and cooled. A part of this residue is preserved for flame test. The remaining part of the residue is dissolved in about 2ml of water. It is divided into two parts and tested as below.

<p>Test for Na^+: I part + alc.KOH + + 10 drops of potassium pyro-antimonate solution, the inner sides of the test tube is scratched with the help of a glass rod.</p> <p><u>Confirmatory test:- Flame test</u> Above step residue or II part of the evaporated solid extract + a drop of Con. HCl, flame test is conducted</p> <p>Test for K^+: II part +10 drops of Picric acid solution, the inner sides of the test tube is scratched with the help of a glass rod.</p> <p><u>Confirmatory test:- Flame test</u> Above step residue or II part of the evaporated solid extract + a drop of Con. HCl, flame test is conducted.</p>	<p>White precipitate</p> <p>Golden Yellow flame</p> <p>Yellow crystalline precipitate</p> <p>Violet or lilac colored flame</p>	<p>Na^+ is present</p> <p>Na^+ is confirmed</p> <p>K^+ is present</p> <p>K^+ is confirmed</p>
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Ionic reactions: 1. $2\text{Na}^+ + \text{Sb}_2\text{O}_7^{4-} + 2\text{H}^+ \rightarrow \text{Na}_2\text{H}_2\text{Sb}_2\text{O}_7$ (White ppt.)

2. $\text{K}^+ + \text{C}_6\text{H}_2(\text{NO}_2)_3\text{OH} \rightarrow \text{H}^+ + \text{C}_6\text{H}_2(\text{NO}_2)_3\text{O}^- \text{K}^+$ (Yellow ppt.)

Report: The given salt mixture contains:

Acid radicals		
Basic radicals		

SYSTEMATIC SEMI-MICRO QUALITATIVE ANALYSIS OF A SALT MIXTURE CONTAINING TWO ACID AND TWO BASIC RADICALS

Model procedure for the given analysed salt

- 1. Preliminary Tests:** i) State: Solid, ii) Appearance: Amorphous
iii) Color: Colorless iv) Solubility: Soluble in dil. HCl

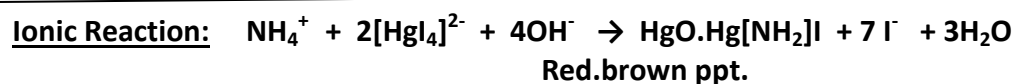
2. Detection of Acid Radicals:

Experiment	Observation	Inference
<p>Salt mixture + Dil.HCl</p> <p>Nature of the evolved gas The vapours liberated gas is passed into a test tube containing lime water</p> <p><u>Confirmatory test for $\text{CO}_3^{2-}/\text{HCO}_3^-$</u> Salt mixture + water, boiled, the liberated vapours are passed into a test tube containing lime water</p> <p>Salt mixture + Con. Sulphuric acid in a dry test tube. Color and nature of the gas A glass rod dipped in ammonium hydroxide is exposed to the out-coming gas.</p> <p align="center"><u>Confirmatory test for chloride: (Chromyl chloride test) :</u></p> <p>Salt Mix. + Potassium dichromate crystals are taken in a dry test tube + con. H_2SO_4 the contents are heated, the red vapours liberated are passed into a test tube containing water + NH_4OH + Acetic acid + Lead acetate, shaken well.</p>	<p>Brisk effervescence</p> <p>Colorless odourless gas</p> <p>Lime water turns milky</p> <p>Lime water does not turns milky</p> <p>Vigorous reaction Colorless fuming gas</p> <p>Dense white fumes</p> <p>Bright yellow precipitate</p>	<p>I Group acid radical is present</p> <p>CO_3^{2-} or HCO_3^- is present.</p> <p>Carbonate(CO_3^{2-}) is confirmed</p> <p>II Group acid radical is present May be chloride</p> <p>Cl^- is present</p> <p>Cl^- is Confirmed</p>

3. Detection of Basic Radicals:

Experiment	Observation	Inference
<p align="center"><u>TEST FOR AMMONIUM RADICAL(NH_4^+)</u></p> <p>5-10mg of the salt mixture + 1ml of NaOH solution, the contents of the test tube is heated. A moist red litmus paper is exposed to the out coming gas</p>	<p>Pungent odoured gas is liberated</p> <p>Red litmus turns blue</p>	<p>VI Group basic radical is present</p> <p>May be Ammonium radical Ammonium radical is present(NH_4^+)</p>

Confirmatory test-Nessler's reagent test : 10mg of the salt mixture + 1ml of sodium hydroxide solution, boiled and the vapours are passed into a test tube containing Nessler's reagent	Reddish brown precipitate	Ammonium is Cofirmed(NH ₄ ⁺)
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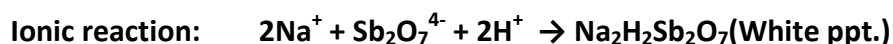


1 ml of the original solution + 1ml of dil HCl in excess	No white precipitate	I group basic radical is absent
About 1ml of original solution + dil HCl + H ₂ S gas is passed or thioacetamide solution dilute with 2-3 drops of water.	No precipitate	II group basic radicals is absent)
Original solution + solid NH ₄ Cl till saturated +excess of liquor ammonia till it smells sufficientl	No precipitate	III group basic radicals is absent

Original solution + Solid NH ₄ Cl + Excess of liquor ammonia soln.+ H ₂ S gas is passed or thioacetamide solution is added	No precipitate	IV Group basic radical is absent
Original solution + Solid NH ₄ Cl + Excess of liquor ammonia soln + (NH ₄) ₂ CO ₃ solution.	No precipitate	V Group basic radical is absent
Original solution is divided into two unequal parts and tested as follows.		
Test for Mg²⁺: Smaller part+solid NH ₄ Cl + Excess of liquor NH ₃ +10 drops of ammo- nium hydrogen Phosphate solution, shaken well.	No precipitate	Mg ²⁺ is absent

Tests for Na⁺ and K⁺: Larger part of the Original solution+ 1-2 drops of con.HCl and the solution is evaporated to dryness in a small porcelain crucible with stirring till no more fumes are liberated and cooled. A part of this residue is preserved for flame test. The remaining part of the residue is dissolved in about 2 ml of water. The solution is divided into two parts and tested as below.

<p>Test for Na⁺: I part + alc.KOH + + 10 drops of potassium pyro-antimonate solution, the inner sides of the test tube is scratched with the help of a glass rod.</p> <p>Confirmatory test:- Flame test Above step residue or II part of the evaporated solid extract + a drop of Con. HCl, flame test is conducted</p>	<p>White precipitate</p> <p>Golden Yellow flame</p>	<p>Na⁺ is present</p> <p>Na⁺ is confirmed</p>
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Report: The given salt mixture contains

Acid radicals	Carbonate (CO ₃ ²⁻)	Chloride(Cl ⁻)
Basic radicals	Ammonium(NH ₄ ⁺)	Sodium(Na ⁺)

PROCEDURE WRITING EXPERIMENTS

1. SEPARATION OF Fe²⁺ AND Mg²⁺ FROM A MIXTURE BY SOLVENT EXTRACTION METHOD

Principle: Solvent extraction process is based on the Nernst distribution law

$$\text{Partition coefficient (K)} = \frac{\text{Concentration of solute in organic layer}}{\text{Concentration of solute in aqueous layer}}$$

Higher the partition coefficient more is the solubility of the solute in the organic layer. Chelate metal complex is more soluble in organic layer.

Procedure: The mixture containing Fe²⁺ and Mg²⁺ ions is treated with calculated quantity of H₂O₂ in the presence of dilute H₂SO₄. Fe²⁺ is oxidized to Fe³⁺. 50cm³ of this solution is taken in a separating funnel, and 10 cm³ of 1% oxine solution (8-hydroxy quinoline) in chloroform is added, maintaining the pH between 2-3. Only Fe³⁺ ions form a complex with oxine under these conditions, and this complex dissolves in the chloroform layer Mg²⁺ ions remain in the aqueous layer. The organic layer is removed from the separating funnel. The complex is now decomposed using HCl, and the Fe³⁺ ions are recovered. The aqueous layer is separated which mainly contains Mg²⁺ ions.

Note: K for the oxinate complex between CHCl₃ and water is about 720

2. ESTIMATION OF C.O.D IN THE GIVEN SAMPLE OF EFFLUENT

Principle: The amount of organic waste present in a sample of water is expressed in terms of C.O.D It is defined as the amount of oxygen in mg/L required to completely oxidize the oxidizable organic matter. Higher the C.O.D. more polluted is the water.

Procedure: 25 cm³ of the given effluent sample is taken in a round bottomed flask fitted with reflux condenser. 10 cm³ of 0.25N K₂Cr₂O₇ solution and a small quantity of solid HgSO₄ and Ag₂SO₄ are added to it. 20 cm³ of concentrated H₂SO₄ is then added. The resulting mixture is refluxed for about 2 hours. The contents of the flask are diluted with water and a few drops of ferroin indicator is added. The mixture is titrated with 0.1N Mohr's salt solution till the colour changes from blue to red. The titre value is noted(A). A blank titration is carried out using 25 cm³ of distilled water instead of effluent. The blank titre value is also noted(B).

Calculation: Titre value for experimental solution = A

Blank titre value = B

Volume of Mohr's salt used by excess K₂Cr₂O₇ = (B - A) cm³

1N K₂Cr₂O₇ = 1N FAS = 8g of oxygen

1000cm³ of 1N FAS = 8000mg of O₂

$$(B - A) \text{ cm}^3 = \frac{B - A}{1000} \times 8000 \text{ mg of O}_2$$

V cm³ of effluent = 8 (B - A) mg of O₂

$$1000 \text{ cm}^3 \text{ of effluent} = \frac{8 (B - A)}{V} \times 1000 \text{ mg of O}_2$$

$$\text{C.O.D in mg/liter} = \frac{(B - A) \times 8000N}{1000} \quad (N \text{ is the normality of FAS})$$

Result: The C.O.D of the given sample is _____ mg.