

MIXTURE ANALYSIS

HOW TO ANALYSE A MIXTURE

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WHAT IS AN INORGANIC MIXTURE?

INORGANIC MIXTURE is a mixture of two or more salts. For BSc. Practicals you are given a mixture of two salts

Means it contains 2 anions and 2 cations

Out of 2 anions one may be or may not be interfering .

No 2 cations of same group will be given.

Group reagents

1 Group - Dil.HCl

2 Group – Dil.HCl and H₂S

3 Group – NH₄Cl and NH₄OH

4 Group - NH₄Cl, NH₄OH and H₂S

5 Group - NH₄Cl, NH₄OH and (NH₄)₂CO₃

6 Group – NH₄Cl, NH₄OH and Na₂(HPO₄)

Group cations

- 1 group- Pb^{2+}
- 2 group- Cu^{2+} , cd^{2+} , Bi^{3+} ,
- 3 Group- Al^{3+} , Fe^{2+} , Fe^{3+} ,
- 4. Group- Co^{2+} , Ni^{2+} , Mn^{2+} , Zn^{2+}
- 5. Ba^{2+} , Sr^{2+} , Ca^{2+}
- 6. Mg^{2+} , Na^{+} , K^{+}
- 0- NH_4^{+}

ANIONS TO BE ANALYSED

Carbonate, acetate, oxalate,
chloride, Fluoride, borate, sulphate.

PROCEDURE OF MIXTURE ANALYSIS

- 1. Grind the given mixture in a mortar.
- 2. Analyse Anions
 - 2.1 Dry tests
 - 2.2 Wet tests
- 3. Eliminate acid radical (anions) if there is
- 4. Test for ammonium
- 5. Analyse cations
 - 5.1 Intergroup separation.
 - 5.2 Group analysis
- 6. Correct reporting

SCHEME FOR ANALYSIS OF INORGANIC MIXTURES.

PRELIMINARY EXAMINATIONS			
1.	Colour of the mixture is noted	Green	Cu ²⁺ or Ni ²⁺ may be present
		blue	Cu ²⁺ may be present
		Light pink	Mn ²⁺ may be present
		pink	Co ²⁺
		colourless	Absence of Cu ²⁺ , Ni ²⁺ , Co ²⁺ , Fe ²⁺ Fe ³⁺ etc.
2.	solubility		
	water	soluble	Water soluble salts are present
	Dil.HCl	Insoluble	May be presence of 1gp cations
3.	Flame test- a little of the mixture is mixed with conc.Hcl and shown to a nonluminous flame using a	Pale green colour.	May be presence of Barium.
		green	May be borate.
		Blue or bluish	May be copper.

Preliminary examination continued

<p>4. Flame test with H_2SO_4 and CaF_2 A little of the mixture is mixed with CaF_2 and conc. H_2SO_4 in a watch glass, made into a paste with glass rod and shown to nonluminous flame</p>	<p>A green flame</p>	<p>Presence of borate is identified- The volatile BF_3 imparts green colour to the flame.</p>
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TESTS FOR AMMONIUM ION

<p>1. A little of the mixture is boiled with NaOH solution and a glass rod dipped in Conc. HCl is shown to the issuing vapours</p>	<p>A gas with the smell of ammonia which gives dense white fumes with a glass rod dipped in conc. HCl</p>	<p>Presence of ammonium is identified- the evolved gas is ammonia which gives dense white fumes of NH_4Cl with HCl.</p>
<p>2. A little of the mixture is boiled with NaOH solution and a filter paper soaked in</p>	<p>A brown stain on the filter paper</p>	<p>Presence of ammonium is identified. The brown stain is due to basic mercuric</p>

ANALYSIS OF MIXTURE

Analysis of anions

Dry tests

1	Action with dil HCl	•Brisk effervescence with the evolution of colourless odour less gas turning lime water milky	Presence of Carbonate is identified .The evolved gas is CO ₂ .Which reacts with Ca (OH) ₂ forming CaCO ₃ .
2	Action with Dil.H ₂ SO ₄	Vinegar smell	Presence of acetate is identified. Vinegar smell is due the formation of acetic acid
3	Action with dil.H ₂ SO ₄ + MnO ₂	Brisk effervescence with the evolution of colourless odour less gas turning lime water milky	Presence of oxalate is identified .The evolved gas is CO ₂ .Which reacts with Ca (OH) ₂ forming CaCO ₃ .
4.	Action with conc.H ₂ SO ₄	<ol style="list-style-type: none">1. Colourless fuming gas, white coating on a moist glass rod, oily appearance in the test tube.2.A colourless fuming gas which gives dense white fumes when a glass rod dipped in amm.hydroxide is showed in it.3.Reddish brown vapours.4.Violet vapours condensing to black solid on the cooler sides of the test tube	<p>presence of fluoride is identified.The evolved gas is Hf which reacts with glass forming SiF₄.</p> <p>Presence of chloride is identified. The evolved gas is HCl.which reacts with ammonia giving dense white fumes of NH₄Cl.</p> <p>Presence of Br- is identified. Bromine gas is produced.</p> <p>Presence of I- identified. The violet vapours are due to iodine gas.</p>
5	Conc.H ₂ SO ₄ and MnO ₂	<ol style="list-style-type: none">1.A greenish yellow gas2.Reddish brown vapours.3. Violet vapours condensing to black solid on the cooler sides of the test tube.	<p>Presence of chloride is identified. The evolved gas is chlorine.</p> <p>Presence of bromide is identified. The evolved gas is bromine.</p> <p>Presence of I- identified. The violet vapours are due to iodine gas.</p>

Anion analysis continue... . . .

6.	Conc.H ₂ SO ₄ + paperball	Plenty of reddish brown gas is evolved.	Presence of NO ₃ ⁻ . Nitric acid formed is decomposed by carbon to yield reddish brown nitrogen dioxide.
7.	Chromyl chloride test A little of the mixture is mixed with pot.dichromate And warmed with conc. H ₂ SO ₄ . The above vapours are passed through NaOH solution. The solution is treated with acetic acid and lead acetate solution	A greenish brown gas A yellow solution. A yellow precipitate.	Presence of chloride. Chromyl chloride vapours are produced. This is due to the formation of sodium chromate. The precipitate is lead chromate.
8.	Ethylborate test	A green edged flame	Presence of borate is confirmed. Volatile ethyl borate imparts a green colour to the flme.
9.	Ethylacetate test	A pleasant fruity smell.	Presence of acetate is confirmed. The fruity smell is due to ethyl acetate.
10.	Ammonium molybdate test	A canary yellow precipitate is formed	presence of phosphate is identified. The precipitate is due to ammonium phosphomolybdate.

Sod. Carbonate Extract

1	Dil. HNO ₃ + AgNO ₃	<p>Curdy white precipitate soluble in NH₄OH .</p> <p>A pale yellow precipitate partially soluble in NH₄OH.</p> <p>A yellow precipitate insoluble in NH₄OH</p>	<p>Presence of Cl⁻. The precipitate is silver chloride which dissolves in NH₄OH forming Ag(NH₃)₂Cl.</p> <p>Presence of bromide is confirmed. The precipitate is Ag Br.</p> <p>Presence of iodide is confirmed. The precipitate is AgI</p>
2	Dil. HNO ₃ + AgNO ₃ + NH ₄ OH	A yellow precipitate	Presence of phosphate. The yellow precipitate is silver phosphate.
3	Dil. HCl + BaCl ₂	A white precipitate soluble in Conc. HCl.	Presence of sulphate is confirmed. The precipitate is Ba SO ₄ .
4	Acetic acid + CaCl ₂	A white precipitate	Presence of fluoride or oxalate. - The white precipitate is due to calcium fluoride or calcium oxalate
	The above precipitate is dissolved in dil. H ₂ SO ₄ and a few	<p>KMnO₄ is decolourised.</p> <p>KMnO₄ is not decolourised</p>	<p>Presence of oxalate is confirmed. - Permanganate is reduced to colourless Mn²⁺.</p> <p>Presence of fluoride is confirmed. -</p>

INTERGROUP SEPARATION

Dilute hydrochloric acid is added to the solution and filtered/centrifuged.

Residue -
Presence
of Group 1
cations

Centrifugate: It is diluted, heated and then H₂S is passed through it. Centrifuged.

Residue:
Presence
of group II
cations

Centrifugate: H₂S is boiled off. then excess Ammonium chloride and ammonium hydroxide are added. Centrifuged.

Residue:
Presence
of group
III
cations

Centrifugate: Concentrated . excess ammonium chloride and ammonium hydroxide are added. H₂S gas is passed. Centrifuged.

Residue:
Presence
of group
IV cations

Centrifugate: Concentrated then excess ammonium chloride , ammonium hydroxide and ammonium carbonate solution are added. Centrifuged.

Residue:
Presence
of group

Centrifugate:
Tested for Vi group
cations nd found to be

ANALYSIS OF GROUP I RESIDUE

The residue is boiled with water

No Residue
–Absence
of Hg^+ and
 Ag^+

Centrifugate- The hot solution is divided into two.

1.To one portion acetic acid and potassium chromate added- yellow residue-presence of lead is identified-the yellow residue is due to lead chromate.

2.The second portion is treated with potassium iodide-A yellow precipitate which dissolves on heating with water, but reappears on cooling in the form of golden spangles- Presence of lead is confirmed-The yellow precipitate is due to the formation of lead iodide.

ANALYSIS OF GROUP II RESIDUE

The group II residue is boiled with dilute nitric acid and centrifuged.

No residue absence of mercuric ion	Centrifugate- treated with excess of dilute sulphuric acid and centrifuged.		
	No black Residue . absence of lead	centrifugate -concentrated and treated with Ammonium hydroxide. A white Residue presence of Bismuth is identified-The residue is due to Bismuth hydroxide.- $\text{Bi}(\text{OH})_2$. Dissolved in dilute HCl and divided into two portions 1.To one portion large excess of water is added-White turbidity - presence of Bismuth is identified-The white turbidity is due to the formation of Bismuth oxy chloride by the hydrolysis of Bismuth chloride.- BiOCl 2.The second portion is treated with sodium stannite solution-Black precipitate -Presence of	Centrifugate-No blue colour- absence of copper 1. H_2S is passed to a portion of the centrifugate -yellow precipitate presence of cadmium confirmed- the precipitate is due to cadmium sulphide.- CdS .

ANALYSIS OF GROUP III RESIDUE

Residue is mixed with sodium hydroxide

No residue -absence of iron.

1 .Boiled solid Ammonium Chloride-A white gelatinous precipitate- presence of aluminium is identified-The precipitate is Aluminium hydroxide.- $\text{Al}(\text{OH})_3$.

2.The precipitate is boiled with a little concentrated nitric acid and a few drops of Cobalt Nitrate solution-A piece of filter paper is soaked in the solution and burnt to ashes- a blue tinted Ash – Presence of aluminium confirmed -This is due to the formation of double oxide of aluminium and Cobalt ($\text{Al}_2\text{O}_3 \cdot \text{CoO}$. Thenard's blue).

ANALYSIS OF GROUP IV RESIDUE

Group 4 residue is shaken with very dilute hydrochloric acid and centrifuged

Black Residue- presence of Nickel or Cobalt.

The Residue is dissolved in Aqua regia (1:3 HNO₃:HCl) in a china dish and evaporated to dryness.-

A yellow residue- Presence of Ni²⁺. Is identified- The yellow residue is NiCl₂'

A blue residue- Presence of Co²⁺ is identified-The blue residue is CoCl₂.

The residue is dissolved in water and divided into 2 portions.

1.To one portion NH₄Cl, NH₄OH and potassium ferricyanide solution are added- a reddish brown precipitate- Presence of Cobalt is confirmed- the precipitate is cobalt ferricyanide.Co₃[Fe(CN)₆]₂.

Centrifugate-H₂S is boiled off. Excess of NaOH is added and boiled.

1.A white residue gradually turned to brown - Presence of Mn²⁺- is identified-White Mn(OH)₂ is formed which undergo atmospheric oxidation to brown hydrated manganese dioxide(MnO(OH)₂).

2.A very small of the residue is mixed

Centrifugate- Divided into 2 portions

1. H₂S is passed through one portion- white residue-Presence of Zinc is identified-The residue is due to ZnS

2. 2. The residue is mixed with cobalt nitrate , nitric acid , boiled. The filter paper is soaked in it and burnt to ashes.-

ANALYSIS OF GROUP V RESIDUE

The group V residue is boiled with acetic acid and potassium chromate solution is added

Yellow residue- Presence of Barium is identified-The residue is due to the formation of barium chromate- BaCrO_4 .

1. Flame test-One portion of the is dissolved in Conc.HCl and flame test is conducted .A portion of the solution is shown to the nonluminous flame using a glass rod.-A green edged flame- presence of barium is confirmed- This is characteristic of barium salts.(gas excitations gives a characteristic colour in emission spectrum).

2. One portion of the residue is dissolved in Conc.HCl and dilute sulphuric acid is added – A white precipitate- Presence of barium is again confirmed- The white

Centrifugate is concentrated by evaporation and divided into 2 portions.

1.(A) To one portion dilute sulphuric acid is added- A scanty white precipitate- Presence of strontium is identified- The precipitate is strontium sulphate- (SrSO_4)

1.(B) Flame test- The precipitate is dissolved in conc.HCl and shown to non luminous flame with help of a glass rod-A crimson red flame- Presence of strontium is confirmed- This is characteristic of barium salts.(gas excitations gives a characteristic colour in emission spectrum).

2 (A) To the second portion of centrifugate

ANALYSIS OF GROUP VI RESIDUE

The filtrate for VI group analysis is evaporated in a china dish nearly to dryness added 5 ml conc. HNO_3 again evaporated to dryness. dissolved in water and the following tests are conducted.

1. sat. $\text{NH}_4\text{Cl} + \text{NH}_4\text{OH} + \text{Na}_2\text{HPO}_4$ added to a portion of the solution, shaken well- A white crystalline precipitate- presence of Mg^{2+} identified- The white precipitate is due to magnesium ammonium phosphate. $\text{Mg}(\text{NH}_4)\text{PO}_4$.
2. The above precipitate is dissolved in conc. HNO_3 , mixed with cobalt nitrate, a piece of filter paper is soaked in the solution and burnt to ashes- a pink tinted ash- presence of Mg^{2+} is confirmed- The pink ash is due to double oxide of $\text{MgO} \cdot \text{CoO}$.
3. To a little of the solution excess of NaOH is added followed by few drops of magesone reagent- A sky blue precipitate- Presence of Mg^{2+} is confirmed- Magnesium hydroxide formed adsorbs the dye to yield a sky blue precipitate.
4. To a portion of the solution equal volume of sodium hydrogen tartrate is added and shaken well.- A white precipitate- Presence of K^+ is identified- The precipitate is Potassium hydrogen tartrate.
5. To a portion of the solution potassium pyroantimonate solution is added- white residue- presence of Na^+ is identified- The precipitate is sodium pyro antimonate.
6. Flame test is conducted using the initial residue by mixing with conc. HCl and showing to non luminous flame using a glass rod-
Lilac flame- Presence of K^+ is confirmed- confirmed- This is characteristic of potassium salts. (gas excitations gives a characteristic colour in emission spectrum).
Golden yellow flame- Presence of Na^+ is confirmed.- confirmed- This is characteristic of sodium salts. (gas excitations gives a characteristic colour in emission spectrum).