Flame test

The substance is moistened with conc. HCl, and the mixture, on a platinum wire is shown in the edge of the non-luminous Bunsen flame.

Flame Colour	Inference
Golden yellow	Na
Lilac (violet)	K
Brick – red	Ca
Crimson	Sr
Apple green	Ba
Bluish green	Cu
Green	Borates
Livid blue	Pb, Sb, Bi

Dil. Acid group

To about 5 mg of the substance add about 0.5 ml of dil. HCl. Observe the reaction in the cold and then heat it on a water bath.

Observation	Inference
1. **Brisk effervesce**, the gas turns lime/Baryta water milky	**Carbonate** is Present.
2. Colourless gas (SO_2) with the smell of burning sulphur .The gas turns a filter paper dipped in acidified potassium dichromate green.	**Sulphite** is Present.
3. Colourless gas with the odour of rotten eggs (H_2S) is evolved. The gas turns lead acetate paper black and cadmium acetate paper yellow.	**Sulphide is** Present.
4. Vineger smell. Red coloration/ppt with neutral ferric chloride. 5. Mix 20 mg of the substance with 1ml of ethyl alcohol and 5 drops of con. H_2SO_4, Heat in a hot water rack for 10 minutes, and pour into 2 ml of Na_2CO_3 solution. → Fruity odour	**Acetate** is Present.

Conc. Acid group

Addition of Conc. H_2SO_4 + MnO_2, heat

Observation	Inference
1. **Greenish yellow** pungent smelling gas (HCl) which fumes in moist air. Dense white fumes (NH_4Cl) with a drop of ammonia on a glass rod	**Chloride** is Present.
2. **Reddish brown** fumes (Br_2) are evolved.	**Bromide** is Present.
3. **violet** vapours (I_2) are evolved.	**Iodide** is Present.
4.On warming, **brown** gas (NO_2)With characteristic smell is evolved. The brown colour is deepened by the addition of copper turnings.	**Nitrate** is Present.

Silver nitrate group

Add silver nitrate to Neutralized sodium carbonate extract

1	A curdy white precipitate (AgCl), insoluble in dil.HNO_3, but soluble in ammonia solution.	**Chloride** is confirmed.
2	A pale yellow precipitate (AgBr), insoluble in dil. HNO_3, but sparingly soluble in ammonia solution.	**Bromide** is confirmed.
3	A yellow precipitate, insoluble in both dil. HNO_3 and ammonium solution.	**Iodide** is confirmed.

Test for Chloride
Chromyl chloride test

Test for Bromide and Iodide:

To the substance in dil. HNO_3 add drops of $KMnO_4$ solution until the pink colour persists. Add CCl_4 and shake.

Reddish brown colouration of CCl_4 layer	Bromide is confirmed.
Violet colouration of CCl_4 layer.	Iodide is confirmed.

Nitrate : Brown ring test

The sodium carbonate extract is acidified with dil. H_2SO_4.

An equal volume of freshly prepared $FeSO_4$ solution is added.

Holding the test-tube in an inclined position con. H_2SO_4 drops are added without shaking.

A **brown ring** is formed at the junction of the two layers	Nitrate is confirmed.

Sulphate : $BaCl_2$ test

To the sodium carbonate extract add dil. HCl till no more CO_2 is evolved. Add 1–2 ml of dil. HCl and $BaCl_2$ solution.

A white precipitate insoluble in dil. HCl is formed	Sulphate is Confirmed

Borate: Flame Test

The substance is mixed with calcium flouride and con. H_2SO_4 to get a paste. Hold some of the paste on a platinum loop, just outside the base of the Bunsen flame.

A **green** flame is formed	Borate is confirmed.

Phosphate : Amm. Molybdate test

To the sodium carbonate extract add dil. HNO_3ill no more CO_2 is evolved. Add 1–2 ml of amm. Molybdate. Warm.

Yellow ppt	Phosphate is confirmed.

Analysis of Cations

- **Preparation of the original solution**

A small quantity of the substance (15 mg) is treated with the following solvents in the given order.

- **Distilled water**
- **dil.HCl,**
- **dil.HNO_3**
- **con.HCl**
- **aqua regia** (3 vol. con. HCl + 1 vol. con. HNO_3).

Observe the solubility in the cold, then heat to boiling. If any gases are formed, boil them off. Dissolve 50 to 100 mg of the substance in the suitable solvent and prepare the solution. This solution is often referred to as the original solution.

Separation of group 1 cations: The Residue-1 is washed with cold water containing a few drops of dil. HCl, and centrifuged. To the residue, add 1 ml of hot water. Heat to boiling for 1-2 minutes. Centrifuge while hot. Transfer the centrifugate quickly to another test tube.

Residue (Residue 1.1): White: May contain Hg_2Cl_2 and AgCl. Wash with boiling water to remove the undissolved $PbCl_2$.Treat the residue with 0.5 ml warm dilute NH_3 solution. Stir. Centrifuge.

Residue (1.2), Black:Hg + Hg (NH_2) Cl. Hg_2^{2+} present. Dissolve the ppt. in aqua-regia, heat, divide into two parts.

1. Add stannous chloride - White grayish ppt. Hg_2^{2+} is confirmed.

2. Add drops of KI solution-- Red or Yellow ppt. Hg_2^{2+} is confirmed.

Centrifugate (1.2): May contain Ag $(NH_3)_2Cl$. Divide into 2 parts.

1. Add dil.HNO_3. White ppt. (AgCl) – Ag is confirmed.

2. Add KI solution- Yellow ppt. **(**AgI**)** – Ag is confirmed.

Centrifugate (1.1) : May contain $PbCl_2$. Divide into 3 parts.

1. Cool under tap – White ppt. reappears **.** – Pb^{2+} is confirmed.

2. Add 2 drops of potassium chromate – Yellow ppt. **(**$PbCrO_4$**).-** Pb^{2+} is confirmed.

3. Add 2 drops of KI solution –Yellow ppt. **(**PbI_2**).**Boil the ppt. with water and a few drops of acetic acid and cool. The ppt. dissolves on heating and reappears as golden spangles on cooling – Pb^{2+} is confirmed.

Cations/ Groups / Group reagents

Group	Group reagent	Cations	Ppt formed
I	Dil. HCl	Pb^{2+}, Ag^{1+}, Hg^{1+}	Chlorides
II	Dil. HCl + H S	Pb^{2+}, Bi^{3+}, Cd^{2+}, Cu^{2+}, Sn^{2+}, As^{3+}, Sb^{3+}	Sulphides
III	NH Cl + NH OH	Fe^{3+}, Al^{3+}, Cr^{3+}	Hydroxides
IV	NH Cl + NH OH + H S	Co^{2+}, Ni^{2+}, Zn^{2+}, Mn^{2+}	Sulphides
V	NH Cl + NH OH + NH CO	Ca^{2+}, Ba^{2+}, Sr^{2+}	Carbonates
VI	No group reagent	Mg^{2+}, NH^{+}, K^{+}	--

Separation of Cations into Groups

To 1 ml of the original solution in a centrifuge tube, dil. HCl is added until precipitation, if any, is complete Centrifuge.

Residue -1
White, may contain $PbCl_2$, Hg_2Cl_2 or AgCl.
Group-1 present

Centrifugate-1 Heat on a water bath; pass H_2S gas until the precipitation is complete. Centrifuge.

Residue-2
May Contain
Black:HgS, PbS, CuS.
Brown:Bi_2S_3.
Yellow:CdS, Sb_2S_3,SnS_2
Group – 2 present

Centrifugate-2 : (Eliminate the interfering anions if necessary.Boil off H_2S Add 3 drops of con.HNO_3 and boil.Add 100 mg solid ammonium chloride, heat on a water bath. Add ammonia solution till alkaline, and add 2 drops excess. Warm. Stir. Centrifuge.

Residue-3:
May contain
Reddish-brown:$Fe(OH)_3$
Green:$Cr(OH)_3$
White:$Al(OH)_3$
Group 3 Present

Centrifugate-3: Add 2 drops of NH_3 solution. Warm. Pass H_2S to complete precipitation. Centrifuge. Wash the residue

Separation of Cations into Groups Contd….

<table>
<tr><td rowspan="2">Residue-4:
May contain
Black:
CoS, NiS
Pink: MnS,
White:
ZnS
Group 4 present</td><td colspan="2">Centrifugate-4: Place in a china dish. Acidify with dil. Acetic acid. Evaporate to a pasty mass. Add 5 drops of con.HNO_3.Heat to dryness (till fumes stop)[7] Dissolve the residue in 5 drops of dil. HCl and 1 ml water. Add (in test-tube) 5 drops of 20% NH_4Cl. Add NH_4OH with shaking till alkaline. Add excess of 10% $(NH_4)_2CO_3$ soln. Warm at 50-60^oC. Centrifuge. Wash.</td></tr>
<tr><td>Residue -5
May contain
White:
$BaCO_3$,$SrCO_3$,
$CaCO_3$
Group 5 present</td><td>Centrifugate-5:
Evaporate to a pasty mass, add 0.5 ml con.HNO_3.
Heat to dryness
White residue-
Group-6 present</td></tr>
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