KONGUNADU ARTS AND SCIENCE COLLEGE

(AUTONOMOUS)
Re-accredited by NAAC with A+ Grade - 4th cycle, College of Excellence - UGC
COIMBATORE -29.

DEPARTMENT OF CHEMISTRY

Supported by : DBT STAR College Scheme

LAB MANUAL & SOP
(STANDARD OPERATING PROCEDURE)

CORE PRACTICAL- I : INORGANIC QUALITATIVE ANALYSIS AND PREPARATION

Name : ……………………………………………………….

Class : ……………………………………………………….

Roll No. :……………………………………………………….

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Title : Core Practical - I Inorganic qualitative analysis and Preparation

Subject Code : 25UCH2CL

Name :

Register Number :

E. No	Experimental Details	Issue date	Completed Date	Marks	Signature of the Staff

GENERAL INSTRUCTIONS

1. Before you start an experiment, make adequate preparation try to understand the theory behind it.
2. Start an experiment only atter making sure that all the requirements are available.
3. Provide yourself with a box of matches and sone towels.
4. Wear a lab coat while working in the laboratory.
5. Avoid talking in the laboratory, concentrate on the experiment.
6. If in trouble or in doubt, consult your teacher.
7. Use clean apparatus, keep the table and work book neat and tidy.
8. Keep the reagents in their proper places alter use. Do not interchange the stoppers of reagent bottles.
9. Use the minimum quantity of chemicals for doing any reaction.
10. Never add water to concentrated sulphuric acid. Always add acid to water.
11. Do not pour hot concentrated acid into the sink; cool, dilute with water and then pour into the sink.
12. Do not throw solid waste materials into the sink, but throw them into the waste box.
13. As soon as your practical work is over, close gas tap and water tap. Leave th apparatus clean.
14. Report breakage, if any, to Lab attendant.
15. All tests should be done systematically in the given order since the order in which the tests are done is very important.
16. Tests for ammonium ion should be done using the original solution before any group tests. This is because ammonium chloride and hydroxide are added during group separation; so, ammonium ions will always be present in the later stages of analysis.
17. Groups are identified by the formation of a precipitate on adding the reagents for that group. The colour of the precipitate may provide a hint about the identity of the cation and therefore should be noted. i.e., instead of recording the observation simply as “a precipitate is formed”, write “a white precipitate” is formed or “a green precipitate is formed” etc.
18. In each group, precipitation must be completed by adding excess of the group reagent. Add the reagent drop by drop with shaking, till excess. The precipitate must be completely removed by filtration or centrifugation and the filtrate/centrifugate

concentrated to half its volume before proceeding to the next group. This is because each addition of group reagent will dilute the original solution.

19. The precipitate should be washed well as recommended before proceeding with confirmatory tests. The precipitate can be washed in the centrifuge tube itself by stirring thoroughly with the wash liquid, centrifuging again and discarding the supernatant.
20. When testing for a subsequent group, ions of all previous groups must be totally absent in the mixture. Therefore, if you have gone wrong and must repeat the tests with fresh mixture, you have to start from the beginning each time; analysis cannot be continued from the point of mistake.
21. Using 1 ml of the given mixture, carry out the group tests alone to identify which groups are present. Only the reagents for groups which are present needs to be added to the bulk of the mixture, thus saving time and reagents. Carry out the analysis using half of the mixture given to you, reserving the other half in case you go wrong. Using too little of the mixture will not give sufficient precipitate for identification and confirmatory tests. About 5 ml of the mixture should be sufficient for complete analysis.
22. After each test, also note down the chemistry of the reaction or the specific nature that helped identification of the cation. This will fetch you more marks, create a favourable impression, help you during the viva voce examination and improve your understanding of chemistry.
23. Use only distilled water for dilution and washing in all tests.
24. Record all observations, in your rough record book. Submit it to our teacher and get I signed before you leave the laboratory.
25. Maintain your observation books and fair records neatly, with appropriately number and dated entries. The condition of the records reflects the personality of the student and will influence his internal assessment marks. You must have your observation books during all laboratory sessions. Get them attested by the instructor after completing each experiment.

SAFETY RULES

Most of the chemicals used in the laboratory are toxic. Be aware of the hazards arising out of (i) toxic, corrosive and poisonous chemicals. (ii) broken glass. (iii) explosion (iv) fire and (v) electrical shock.

1. Wear eye -protection at all times inside the laboratory. Even if you wear glasses or contact lens, use goggles while performing unfamiliar reactions.
2. Wear an apron or lab-coat.
3. If corrosive solutions are spilled on clothing, remove the garment immediately.
4. Wear adequate foot covering. Use a net to contain long hair.
5. Avoid wearing flammable clothing.
6. Never touch objects that have been heated (Keep a tube of borneol ointment ready within reach).
7. Always fire polish the ends of freshly cut-glass tubing.
8. Never attempt to force a glass tubing through a hole in a stopper.
9. Use fume hoods (fume cupboards) whenever toxic or noxious gases are likely to be evolved.
10. In the event of burn or injury notify the instructor immediately.
11. Dispose off solutions and chemicals as instructed.
12. Do not perform unauthorised experiments.
13. Never work alone in the laboratory.
14. Never bring food/beverages into the laboratory.
15. Do not drink from laboratory glassware; do not attempt to taste any laboratory chemicals.
16. Do not smoke in the laboratory.
17. Learn the locations of fire extinguishers and learn how to use them.
18. Never leave flames on when not in use.
19. A laboratory is never a place for practical jokes or pranks.
20. It is better to avoid mouth pipetting of liquids; a pipette - filler or other suitable Suction device may be employed.
21. Do not add porcelain chips to a liquid that is already heated to boiling.
22. For alkali metal fire, a bucket of sand is preferable to CO_2 extinguisher.
23. A chemical laboratory is to be equipped with an eye-wash fountain, shower, fire alarm, fire blanket and an emergency exit. You may use them when necessary.

24. Always wash thoroughly any part of your body which comes into contact with any chemical.
25. When carrying materials be sure to avoid overloading.
26. Carry glass tubing's vertically; never horizontally.
27. Know the locations of electrical, gas and water mains.
28. Wear gloves when handling toxic chemicals.
29. Clean all the apparatus used and your work table; wash your hands clean with soap and water at the end of every practical class.
30. Throw all wastes only into the dust - bin provided for this purpose; do not throw them into the sink or elsewhere. Put out the lighted match stick, completely, before throwing it into the dust - bin. Never attempt to light a burner with anything other than match stick or a lighter.
31. While smelling chemicals do not bring them too close to your nose.
32. Any spillage of chemicals on the work bench or on the floor of the laboratory must be leaned immediate is advised by the instructor; broken glass and splinters must be immediately removed and the place swept clean.

FIRST AID

1. **Eye Injuries**: Always wear safety glasses in the laboratory. If any chemical gets into the eyes, the eye irrigated at once with water at the nearest tap or eye bath or with distilled water. Washing should continue atleast for ten minutes. The help of a second person in holding the head and spreading the eye-lids to cause thorough washing is desirable. (Contact of bases with eyes should be taken more seriously). Always only wash with water and not with any other chemical. Seek professional medical attention after prolonged washing (If contact lenses are worn, they should be removed at once).
2. **Fire and Burns**: Ensure that there are no flammable materials in the vicinity before lighting a flame. For could be caused by ineffective destruction of reactive reagents also. For skin burns, cool the affected skin area at once with running water. Larger burns may need to be covered with cloth soaked in water. Seek professional medical attention. Do not cover burns with oily/grease ointments. Do not puncture blisters. Learn the locations of sand buckets, fire extinguishers, fire blankets and fire alarms and learn to use them
3. **Cuts**: Most cuts occur when working with glass. If glass is handled with care, these

accidents can be prevented. Exercise care in pushing glass tubing through holes. Cool ampoules thoroughly before opening them. For small cuts allow to bleed for a while, disinfect and bandage; seek medical attention. For larger cuts may bleeding, cover with bandage and seek medical attention. For cuts on fingers, remove rings if any and go examined by a doctor. To stop bleeding from wounds, raise the affected limb; apply pressure at an appropriate point and apply large dressing; seek medical attention.

4. **Poisoning:** Report the nature of the poison to the doctor. The general procedure is to eliminate the poison obtain medical treatment.

   Oral poisoning: induce vomiting by giving warm salt water (3 teaspoons of NaCl per glass of water, repeat until vomit is clear; seek medical assistance. Do not induce vomiting if unconscious. Do not induce vomiting if solvent has been swallowed.

   If acid has been swallowed, give plenty of water to drink, followed by milk of magnesia; if alkali been swallowed, give plenty of water to drink followed by 1% acetic acid (vinegar).

   In case of gas poisoning, remove victim from danger zone, and transport to doctor, in a horizontal stretcher.

   If breathing has stopped apply artificial respiration, other than mouth to mouth. In acute case factor may be critical.

5. **Conc. H_2SO_4**: Concentrated sulphuric acid spillage on skin should be mopped up with cotton or cloth rapidly as possible before rinsing the area with plenty of water. Then seek medical attention.

6. **Carcinogens**: Working with carcinogens must be avoided. Sometimes the latent period between the chemical insult and the appearance of malignant symptoms could be as long as 40 years in man Given is a partial list of carcinogenic compounds.

   Dimethyl sulphate, $(CH_3O)_2SO_2$, diazomethane, CH_2N_2, iodomethane, CH_3I, Hydrazine, H_2NNH_2, Phenyl hydrazine, $PhNHNH_2$, benzidine, $H_2N.C_6H_4$. $C_6H_4.NH_2$, 2- naphthylamine, Benzene, C_6H_6

   Arsenic compounds, asbestos, nickel and its compounds., chromates, beryllium and its compounds, antimony trioxide, zinc chromate, chromium trioxide, cadmium and its compounds, nitrosamines and thiourea.

7. **Electrical Shock:** In the event of an electrical shock turn off the power supply; remove the electrical contact cautiously from the patient using a dry stick; keep the patient warm using a blanket or a hot water bag: start artificial respiration, if necessary; seek medical assistance.

USE OF LP GAS IN THE LABORATORY – PRECAUTIONS

The liquefied petroleum gas, used in most of the laboratories, is highly inflammable. It should be used carefully to avoid fire accidents.

1. Before opening the burner tap, the match stick should be lighted and kept ready.
2. It is dangerous to light the burner after opening the tap and allowing the unburnt gas to escape for some time
3. Wandering here and there with the lighted match stick is to be avoided. Also lighting one burner from another using paper etc. is not safe.
4. The burners should be put out when not in use.
5. Unburnt gas leaking through burners (or otherwise) is dangerous. The gas taps of such burners should be closed at once and repair work undertaken immediately.
6. The gas taps should never be opened in full; a half - turn opening would often suffice.
7. The table control-valve should not be opened and closed very often.
8. In case of any leakage or in the event of a fire accident, the table control-valve should be closed immediately and the staff informed at once.
9. Avoid pulling or pushing the burners by the rubber tube connection; in case it gets pulled out of the burner, they would cause major damage and fire.
10. The gas cylinders should always be handled by the laboratory staff.
11. In the event of gas leakage anywhere (which could be detected easily by smell), it should be promptly reported to the laboratory staff for remedial measures.
12. Wearing nylon and other inflammable garments should be avoided in the laboratory. Cotton dress is preferable to synthetic fabrics.
13. All students should be conversant with general fire accident precautions and the first aid methods to follow.
14. If you are slack, you will be hurting yourself seriously and, others may also be affected by your carelessness.

GENERAL RULES FOR HANDLING REAGENTS AND APPARATUS

1. Established purity of chemicals is crucial to the performance of successful! analytical work.
2. A freshly opened bottle of a reagent - grade chemical can be used with confidence in most applications.
3. Replace the stoppers/droppers immediately after using the reagent.
4. Hold stoppers of reagent bottles between fingers; never leave them on the table / desktop
5. Never return any excess reagent or solution to a bottle.
6. Allow concentrated acid bottles to stand on porcelain or plastic tray; store concentrated nitric acid in bro bottles.
7. Keep strong alkali bottles on tiles or saucers as they will dissolve wax and varnish on bench tops.
8. Use the fume cupboard when handling bromine, hydrogen sulphide and other such harmful chemicals.

SALT ANALYSIS

Systematic procedure for the Qualitative analysis of a mixture of Inorganic salts

PRELIMINARY TESTS			
S.No.	**EXPERIMENT**	**OBSERVATION**	**INFERENCE**
1.	**Colour:** Colour and Appearance of the substance	**Pale green**	**Ferrous** or **Nickel** may be present.
		Green	**Copper**, **Ferrous**, **Nickel** and **Chromium** may be present
		Blue or **bluish green**	**Copper** may be present.
		Yellow or **Brown**	**Ferric** may be present
		Pink	**Manganese**, **Cobalt** may be present.
		Colourless	Absence of Copper, Ferrous, Ferric, Chromium, Cobalt, Nickel etc.
2.	**Solubility** a). In water	**Soluble**	Presence of **water-soluble salts**.
		Insoluble	Absence of water-soluble salts.
	b). In dil HCl	**Soluble**	Presence of **I Group.**
		Insoluble	Absence of I Group.
3.	**Action of Heat** A small amount of the **substance** was **heated** strongly in a **dry test tube**.	**Reddish brown vapours** with crackling noise.	May be **Nitrate**.
		Charring with the smell of **burnt sugar**.	May be **Oxalate**.
		White sublimate at the sides of the test tube, a **gas** with **ammonia smell**. Turned Red Litmus Paper blue.	May be **Ammonium salts**.
		Yellow when **hot** and **white** when **cold.**	May be **Zinc**.
		Colourless gas turned lime water milky.	May be **Carbonate** and **Oxalate**.
		No characteristic gas	Absence of Lead,

		was evolved.	Ammonium, Zinc, Carbonate, Oxalate and Nitrate salts
4.	**Flame Colour Test:** **Substance** + **Conc. HCl** made into a **paste** in a watch glass. The paste was taken in a **glass rod** and introduced into the **non-luminous flame**.	**Bright green**	May be **Borate**.
		Apple green	May be **Barium**.
		Bluish green	May be **Copper**.
		Brick red	May be **Calcium**.
		Crimson red	May be **Strontium**.
		No characteristic- Coloured flame	Absence of Borate, Barium, Copper, Calcium and Strontium.
5.	**Boron Trifluoride Test:** **Substance** + **Calcium Fluoride** (**CaF_2**) + **Conc. H_2SO_4**, made into a paste in a watch glass. The **paste** was taken in a **glass rod** and introduced into the **non-luminous flame**.	**Green-edged flame**.	Presence of **Borate**.
		No Green-edged flame.	Absence of Borate.
6.	**Ethyl Borate Test** **Substance** + 2mL of **ethyl alcohol** + **Conc. H_2SO_4** taken in a test tube and heated. Then, the **edge** of the **test tube** was shown to **the flame**.	**Green Edge flame**.	**Borate** is confirmed.
		No green edge flame	Absence of Borate.
7.	**Action of dil. HCl:** **Substance** + 3 drops of **dil. HCl** taken in a test tube and heated.	**Brisk effervescence** with a colourless gas turned lime water milky.	**Carbonate** is confirmed.
		No characteristic reaction.	Absence of Carbonate.
8.	**Action of dil. H_2SO_4 and MnO_2 Test** **Substance** + **3 drops of dil. H_2SO_4** taken in a test tube and heated. To the hot solution a pinch of **MnO_2** is added.	**Brisk Effervescence.**	Presence of O**xalate**.
		No effervescence takes place.	Absence of Oxalate
9.	**Action of Conc.H_2SO_4** **Substance** + **3 drops of Conc. H_2SO_4** taken in a test tube and **heated**.	**Colourless pungent smelling gas** gave dense white fumes with **NH_4OH**.	May be **Chloride**.

		Immediate **Reddish brown gas** fumes with **NH_4OH**.	May be **Bromide**.
		Colourless gas giving **white precipitate** with a glass rod dipped in water. Oily appearance at the top of the test tube.	May be **Fluoride**
		Yellowish brown vapour evolves on continued boiling and no action of fluorescence paper.	May be **Nitrates**
		A colourless gas evolves turning lime water milky.	May be **Oxalate**
		No characteristic gas is evolved	Absence of Chloride, Bromide, Fluoride, Nitrate and Oxalate.
10.	**Action of Conc. H_2SO_4 and MnO_2 Test** **Substance + 3 drops of Conc. H_2SO_4** taken in a test tube and heated. To the hot solution a pinch of **MnO_2** is added.	**Greenish yellow gas** was evolved.	May be **Chloride**.
		Reddish brown gas was evolved.	May be **Bromide**.
		Brisk effervescence with Colourless gas evolved turned lime water milky.	May be **Oxalate**.
		No characteristic change.	Absence of Chloride, Bromide and Oxalate.
11.	**Action of Con.H_2SO_4 and Copper Turning Test** **Substance + 3 drops of Conc. H_2SO_4 + Copper turning** (or) / *a small piece of filter paper rolled into a ball* taken in a test tube and heated gently.	**Reddish brown gas** was evolved and then the solution turned blue or green	**Nitrate** is confirmed.
		No reddish-brown gas evolved.	Absence of Nitrate.
12.	**Chromyl Chloride Test** Substance + **$K_2Cr_2O_7$ + 3 drops** of **Conc. H_2SO_4** taken in a test tube and heated.	Reddish orange vapours condensed to red oily drops.	**Chloride** is confirmed.
		No Reddish orange vapours produced.	Absence of Chloride.

13.	**Ammonium Molybdate Test** **Substance + 5 drops of Conc. HNO_3** taken in a test tube and heated. Then, Hot solution is added to 2 drops of ammonium molybdate solution taken in another test tube	A canary **Yellow Precipitate** in the cold.	Presence of **Phosphate**.
		No canary yellow Precipitate	Absence of Phosphate.
14.	**Action of Sodium Hydroxide** **Substance + NaOH** solution taken in a test tube and heated.	Colourless gas with **ammonia smell** giving dense fumes with conc. HCl.	Presence of **Ammonium salts**
		No colourless gas with ammonia smell.	Absence of Ammonium salts

PREPARATION OF SODIUM CARBONATE EXTRACT (Soda Extract):

A small amount of given substance taken in a 50 mL of beaker and mixed with three times of sodium carbonate and one test tube of distilled water. Then the beaker was boiled for 10 min and then filtered using filter paper. The filtrate is known as sodium carbonate extract, which was used for the following reactions.

Note: The residue is kept for the analysis of cations after washing with hot water twice or thrice.

TEST WITH SODIUM CARBONATE EXTRACT			
S.No.	**EXPERIMENT**	**OBSERVATION**	**INFERENCE**
13.	**Silver Nitrate Test:** To about 1 mL of the **soda extract + dil. HNO_3** added drop by drop till the effervescence stops. The contents are heated, cooled and **Silver Nitrate ($AgNO_3$)** solution is added.	**Curdy white Precipitate** completely soluble in **NH_4OH**.	**Chloride** is confirmed.
		Pale yellow Precipitate partly soluble in **NH_4OH**.	**Bromide** is confirmed.
		No characteristic precipitate is formed.	Absence of Chloride and Bromide.

14.	**Barium Chloride Test** To about 1 mL of the **soda extract** + **dil. HCl** added drop by drop till the effervescence stops. The contents are heated, cooled and B**arium Chloride** (**$BaCl_2$**) solution is added.	**White Precipitate insoluble in Conc. HCl** is formed.	**Sulphate** is confirmed.
		No characteristic precipitate is formed.	Absence of Sulphate
15.	**Calcium Chloride Test:** About 2 mL of the **soda extract** is acidified with acetic acid (**CH_3COOH**), boiled with 5 mL of C**alcium Chloride** (**$CaCl_2$**) solution.	**White Precipitate**	Presence of **Oxalate** or **Fluoride**.
		No White Precipitate.	Absence of Oxalate or Fluoride.
	If a **white precipitate** obtained in the above experiment, it is filtered off. The white precipitate is dissolved in 3 mL of **dil. H_2SO_4**, heated and to the clear solution; a dil. solution of **Potassium Permanganate (**$KMnO_4$**)** is added drop by drop.	**Potassium Permanganate** (**$KMnO_4$**) **colour** is **decolourised**	**Oxalate** is confirmed.
		Potassium Permanganate (**$KMnO_4$**) is **not decolourised**.	**Fluoride** is confirmed.
16.	**Brown ring test** About 2 mL of the soda extract is acidified with dil. H_2SO_4 and mixed with 3 mL of freshly prepared **Ferrous Sulphate** (**FeSO4**) solution. **Conc. H_2SO_4** is added along the sides of the tube.	A **brown ring** is formed at the junction of the **two liquids**.	**Nitrate** is confirmed.
		No brown ring is formed.	Absence of Nitrate
17.	**Ammonium Molybdate Test** About 1 mL of the soda extract + few drops of **conc. HNO_3** boiled and cooled. Then it is shaken with 10 mL of **ammonium molybdate** (**$(NH_4)_2MoO_4$**) solution.	A **yellow precipitate** is formed on cold or on gentle warming	**Phosphate** is confirmed.
		No Yellow Precipitate is formed.	Absence of Phosphate

ELIMINATION OF INTERFERING ANIONS

Elimination of Oxalate:

A small portion of the **given substance** is roasted in a **china dish** for **ten minutes**. The **residue** was **cooled** and **dissolved** in **dil. HNO_3** and **centrifuged**. The **centrifugate solution (original solution)** is used for the group analysis.

Elimination of Fluoride and Borate:

Boil the substance with **3 mL** of **Conc. HCl** in a china dish and **evaporated to dryness**. The china dish is cooled and **again boiled** with **3 mL** of **Conc. HCl** and evaporated to dryness. This **process** is **repeated** for **3 times**. Finally, the residue is dissolved in 5 mL of water by boiling and centrifuged. The **residue** (**if it is white**) is **analysed for I group cations** (if the **residue is coloured**, it is **rejected**). The **centrifugate (original solution)** is used for the **detection of cations from II group**.

Elimination of Phosphate:

Method 1: Boil off **H_2S** from **Group II Centrifugate**; add **zirconyl chloride** ($ZrOCl_2$) or **Zirconium nitrate** solution and centrifuge. **Zirconium phosphate** settles at the bottom as **residue**. Use the **centrifugate** for **Group III analysis**.

Method 2: The **centrifugate** from **Group II** was boiled to expel H_2S. Then NH_4Cl and NH_4OH were added and the Precipitate formed was dissolved in minimum quantity of dil. HCl. About 3 mL of dil. acetic acid, 5 mL of ammonium acetate and then neutral ferric chloride was added drop by drop with constant stirring until a yellowish-brown Precipitate was formed. Boiled and centrifuged. The residue was analysed for III group and centrifugate was used for the analysis of basic radicals from IV group onwards.

(Or)

The residue obtained from sodium carbonate extract is used for cation analysis (No need for eliminating the interfering radicals by acid elimination, traditional method)

ANALYSIS OF BASIC RADICALS

SEPARATION OF CATIONS INTO GROUPS

Preparation of Original Solution:

The original solution is obtained by dissolving the salt in any one of the following:

(a) Water (cold or hot)
(b) Dil. HCl (cold or hot)
(c) Conc. HCl (cold or hot)
(d) Dil. HNO_3 (cold or hot)
(e) Conc. HNO_3 (cold or hot)

SEPARATION OF CATIONS INTO GROUPS

S.No.	EXPERIMENT	OBSERVATION	INFERENCE
1	To the eliminated solution or original solution **dil. HCl** was added and centrifuged	White Precipitate - **Pb**, **Ag**, & **Hg** ($PbCl_2$, $AgCl$, Hg_2Cl_2)	Presence of I group cations.
2	To the centrifugate, a drop of hydrogen peroxide (**H_2O_2**) is added, boiled and **Na_2S** (or) H_2S is passed and centrifuged.	Dark brown Precipitate - **Bi** (Bi_2S_3);	Presence of II group cations.
		Black Precipitate - **Cu** (CuS); **Pb** (PbS), & **Hg** (HgS)	
		Yellow Precipitate **Sn** & **Cd** (SnS & CdS);	
		Orange Precipitate -**Sb** (Sb_2S_3)	
3	The centrifugate is boiled well to expel **H_2S** gas. (Eliminate phosphate if present) A drop of **Conc. HNO_3** is added, boiled well and then added a little of **NH_4Cl** + excess **NH_4OH** and centrifuged.	Gelatinous white Precipitate - **Al** ($Al(OH)_3$)	Presence of III group cations.
		Reddish brown Precipitate **Fe** ($Fe(OH)_3$);	
		Pale green Precipitate - **Fe** ($Fe(OH)_2$	
		Green Precipitate - **Cr** ($Cr(OH)_3$)	
4	To the centrifugate, **Na_2S** (or) H_2S is passed and centrifuged.	Black Precipitate - **Co** & **Ni** (CoS & NiS)	Presence of IV group cations.
		Flesh brown Precipitate -**Mn** (MnS)	
		Dirty white Precipitate – **Zn** (ZnS)	
5	The centrifugate is boiled well to **expel H_2S gas**. It is concentrated by evaporation. Then added **NH_4Cl** + **NH_4OH** + **Ammonium carbonate** (**NH_4CO_3**) and centrifuged.	White Precipitate - **Ca**, **Sr** & **Ba** ($CaCO_3$, $SrCO_3$ and $BaCO_3$).	Presence of V group cations.
6	The V group centrifugate is divided into 2 portions. Analysis of VI group cations **Magnesium (Mg).**		

Note: If precipitate appears in a group, the precipitate should be completely removed from solution by adding the corresponding group reagent/ precipitant repeatedly till no more precipitate is formed. Otherwise, precipitation can occur parallely in the following groups creating confusion in differentiating the particular group precipitate.

ANALYSIS OF GROUP I CATIONS (Pb, Ag & Hg)

The first group residue is boiled with water and divided into three parts.

EXPERIMENT	OBSERVATION	INFERENCE
1. One-part original solution is cooled.	Appearance of white Precipitate.	Presence of Lead.
2. Second part, original solution + potassium chromate (**K_2CrO_4**) solution.	Yellow Precipitate	Lead is confirmed.
3. The third part, original solution + potassium iodide (**KI**) solution is added, Water is added to a small portion of the yellow Precipitate, boiled and cooled.	Appearance of golden yellow spangles.	Lead is confirmed.

ANALYSIS OF GROUP II A CATIONS (Hg, Pb, Sb, Bi, Cu & Cd)

To the II group residue, added excess of dilute NaOH, boiled and centrifuged.

Residue: Presence of II-A Group Cations	**Centrifuge:** Added dilute HCl and warmed. If Precipitate is obtained, it is analysed for II-B Group cations. If No Precipitate is obtained, II-B Group is absent.

To the II-A group residue, added dil. HNO_3, boiled for a long time and centrifuged.

<table>
<tr>
<td rowspan="2">**No residue.**
Absence of lead and mercury</td>
<td colspan="2">**Centrifugate:**
(Copper **blue**; Bismuth & Cadmium - Colourless)
Added excess of **NH_4OH**, warmed and centrifuged.</td>
</tr>
<tr>
<td>**Residue:**
Dissolved it in **dil. HCl** and divided into two portions.
a) To one portion, added water. **White turbidity** appeared. Presence of **Bismuth (Bi)**.
b) To another portion, added 3 drops of **10 % solution** of **thiourea**. **Yellow colour solution** confirms the **presence of Bismuth (Bi).**</td>
<td>**Centrifugate:**
(Copper **blue**; Cadmium - Colourless)
Divided into two portions.
a) To one portion, **dil.H_2SO_4** drops are added to make the solution slightly acidic. **Na_2S** (or) H_2S gas is passed. **Yellow Precipitate** confirms the **presence of Cadmium (Cd).**
b) To another portion, **dilute acetic acid** is added to make the solution acidic. **Potassium ferrocyanide ($K_2[Fe(CN)_6)$** is added. **Reddish brown Precipitate** confirms the **presence of Copper (Cu).**</td>
</tr>
</table>

ANALYSIS OF GROUP II-B CATIONS

To the **II-B group residue**, added **Conc. HCl**, boiled carefully, diluted and centrifuged.

Residue:	**Centrifugate:**
Absence of Arsenic.	Divided into two portions. a) To one portion, solid oxalic acid is added to saturation. Diluted and **Na_2S** (or) H_2S gas is passed. **Orange Precipitate** confirms the presence of **Antimony (Sb).** b) To another portion, a pinch of zinc dust is added. Warmed and added 2 drops of **Mercuric Chloride** ($HgCl_2$) solution. **White** or **grey (Hg) Precipitate** confirms the **Presence of Tin (Sn).**

ANALYSIS OF GROUP III CATIONS (Al, Fe, Mn & Cr)

To the **III group residue**, added 2 mL of water and a pinch of **Sodium Peroxide (Na_2O_2)**, stirred, boiled and centrifuged.

Residue:	**Centrifugate:**
1. Dissolved a part of residue in **dil. HCl** and divided into two portions. a) To one portion, added **ammonium thiocyanate** (**NH_4SCN**) solution. **Blood red colour** solution confirms the **presence of Iron (Fe).** b) To another portion, **potassium ferrocyanide ($K_2[Fe(CN)_6$)** is added. **Blue colour solution** confirms the **presence of Iron (Fe).** 2. Another part of the residue is boiled with few drops of **Conc.HNO_3** and a pinch of **Lead dioxide** (**PbO_2**) is added and diluted. **Pink colour** solution confirms the presence of **Manganese (Mn).**	Added **dil. HCl** + **NH_4OH** in excess. **Gelatinous white Precipitate** confirms the **presence of Aluminium (Al).**

ANALYSIS OF GROUP IV CATIONS (Ni, Co, Mn & Zn)

IV group residue is washed with water and centrifuged. The residue is digested with 1 ml of dilute HCl and centrifuged.

Residue:	**Centrifugate:** Boiled, added NaOH and centrifuged.	
	Residue:	**Centrifugate:**
Dissolved in **Conc. HCl** + **Conc. HNO_3** and warmed. Dissolved in water and divided into two portions. a) To one portion, added excess **NH_4OH** + **dimethyl glyoxime**. **Rosy red Precipitate** confirms the **presence of Nickel (Ni).** b) To another portion, **NH_4Cl** + **NH_4OH** + **potassium ferricyanide ($K_3[Fe(CN)_6$)** are added. **Reddish brown Precipitate** confirms the **presence of cobalt (Co).**	1 ml of **Conc. HNO_3** + **sodium bismuthate** (**$NaBiO_3$**) is boiled and added water. Allowed to stand. **Pink colour solution** confirms the presence of **Manganese (Mn).**	Divided into two portions. a) To one portion, passed **H_2S gas** (or) **Na_2S**. **Dirty white Precipitate** confirms the **presence of Zinc (Zn).** b) To another portion, added **dil. HCl** + potassium **ferrocyanide ($K_2[Fe(CN)_6$)**. **White Precipitate** is obtained. **Zinc (Zn) is confirmed.**

ANALYSIS OF GROUP V CATIONS

The Precipitate is washed well with water. The residue is dissolved in warm **dil. Acetic acid (CH_3COOH)** to the solution, 4 or 5 drops of **K_2CrO_4** added, boiled and centrifuged.

Residue: **Yellow residue** shows **Barium**. It is treated with a drop of **Conc. HCl** and **flame test** conducted. **Green flame confirms Barium (Ba).**	**Centrifugate:** Divided into 2 portions. (i) To one portion, **Calcium sulphate** (**$CaSO_4$**) solution is added and boiled. **Scanty white Precipitate** shows **Strontium (Sr)**. (ii) Another portion is boiled with saturated solution of **Ammonium Sulphate** [**$(NH_4)_2SO_4$**]-centrifuged.	
	Residue: **White** residue confirms **Strontium** (**Sr**).	**Centrifugate**: Added NH_4Cl, Aq.NH_3 and ammonium oxalate solution. **White Precipitate** confirms **Calcium (Ca).** Flame test with the above Precipitate conducted. **Brick red colour confirms Calcium (Ca).**

ANALYSIS OF GROUP VI CATIONS (ALKALI GROUP)

The V group centrifugate is divided into 2 portions.

1. To one portion of the centrifugate, added **NaOH** in excess. A **white Precipitate** insoluble in excess of NaOH is obtained. **Magnesium is confirmed (Mg).**
2. To another portion of the centrifugate is evaporated to dryness. A few drops of **Conc. HNO_3** are just added to cover the mass. Evaporated to dryness and cooled. The mass is dissolved in dilute acetic acid.

   To a small portion of this solution, added 2 drops of **NH_4Cl** + 2 drops of **NH_4OH** and few drops of **sodium hydrogen phosphate** (**Na_2HPO_4**). The inner side of the test tube is scratched with a glass rod. **White crystalline Precipitate** confirms the **presence of Magnesium (Mg).**

   To another part, few drops of **dil. HCl** were added followed by few drops of **magneson reagent** and drops of **NaOH** solution. **Blue Precipitate** confirms **Magnesium (Mg).**

ZERO GROUP ANALYSIS

Nessler's test: The mixture of substance is dissolved in water and shakes well and add **Nessler's reagent**, **reddish brown Precipitate** confirms **Ammonium $(NH_4)^+$.**

Report:

The given inorganic mixture contains

(i) **Acid Radicals (Anions):** __________________ and ____________________________

(ii) **Base Radicals (Cations):** ___________________and ____________________________

General solubility rules:

- All nitrates are soluble.
- Practically all sodium, potassium, and ammonium salts are soluble.
- All chlorides, bromides, and iodides are soluble except those of silver, mercury(I), and lead (II).
- All sulphates are soluble except those of strontium, barium, and lead (II), which are insoluble, and those of calcium and silver, which are moderately soluble.
- All carbonates, sulfites, and phosphates are insoluble except those of sodium, potassium, and ammonium.
- All sulfides are insoluble except those of the alkali metals, the alkaline earth metals, and ammonium.
- All hydroxides are insoluble except those of the alkali metals. The hydroxides of calcium, strontium, and barium are moderately soluble. Ammonium hydroxide does not exist; ammonium hydroxide is a misnomer for aqueous ammonia, $NH_3(aq)$.

Why sodium carbonate extract?

Sodium carbonate extract (Soda extract) gives an expedient way of bringing all the anions of the sample into solution. Otherwise, they might be insoluble and couldn't carry out the experiment properly. Also, it prevents the interference of cations.

(i) The preparation of sodium carbonate extract affords a convenient method for bringing the anions of the mixture into solution which were otherwise insoluble with caution of salt.

(ii) It removes the basic radical (usually coloured) which interferes in the usual tests of some of the acidic radicals.

(iii) The residue (can be used for the tests of basic radicals of I to VI groups. Such a solution does not involve the problem of removing interfering radicals like oxalate, fluoride, borate and phosphate.

The chemistry behind interfering radicals:

Interfering radicals are anionic radicals like oxalate, tartrate, fluoride, borate and phosphate. They form complex compounds with third group reagents like ammonium chloride and ammonium hydroxide. Thus, elimination of interfering radicals is necessary before entering in to cationic group separation.

In addition to this, oxalate, tartrate, fluoride, borate, silicate and phosphate of the metals are soluble in acidic medium. If you remember, for 1stand 2nd group analysis medium remains acidic (dilute HCl) that's why they do not interfere then. But for 3rd group analysis the medium becomes alkaline by group reagents ammonium chloride and ammonium sulphide. Here interfering radicals come into action and disturb the solubility product of cations which causes their premature or incomplete precipitation.

In acidic medium these salts produce their corresponding acids like oxalic acid, phosphoric acid, hydrofluoric acid, boric acid and tartaric acid. For example, barium oxalate reacts with HCl and produces oxalic acid.

$\mathbf{BaC_2O_4 + 2HCl \longrightarrow BaCl_2 + H_2C_2O_4}$

These interfering acids are weak acids so they do not dissociate completely and remain in solution in their unionized form. Equilibrium is developed between dissociated and un-dissociated acid. Due to the common ion effect the HCl won't allow to suppress the dissociation of Barium oxalate. $\mathbf{H_2C_2O_4 \rightleftharpoons 2H^+ + C_2O_4^{2-}}$ $\mathbf{HCl \longrightarrow H^+ + Cl^-}$

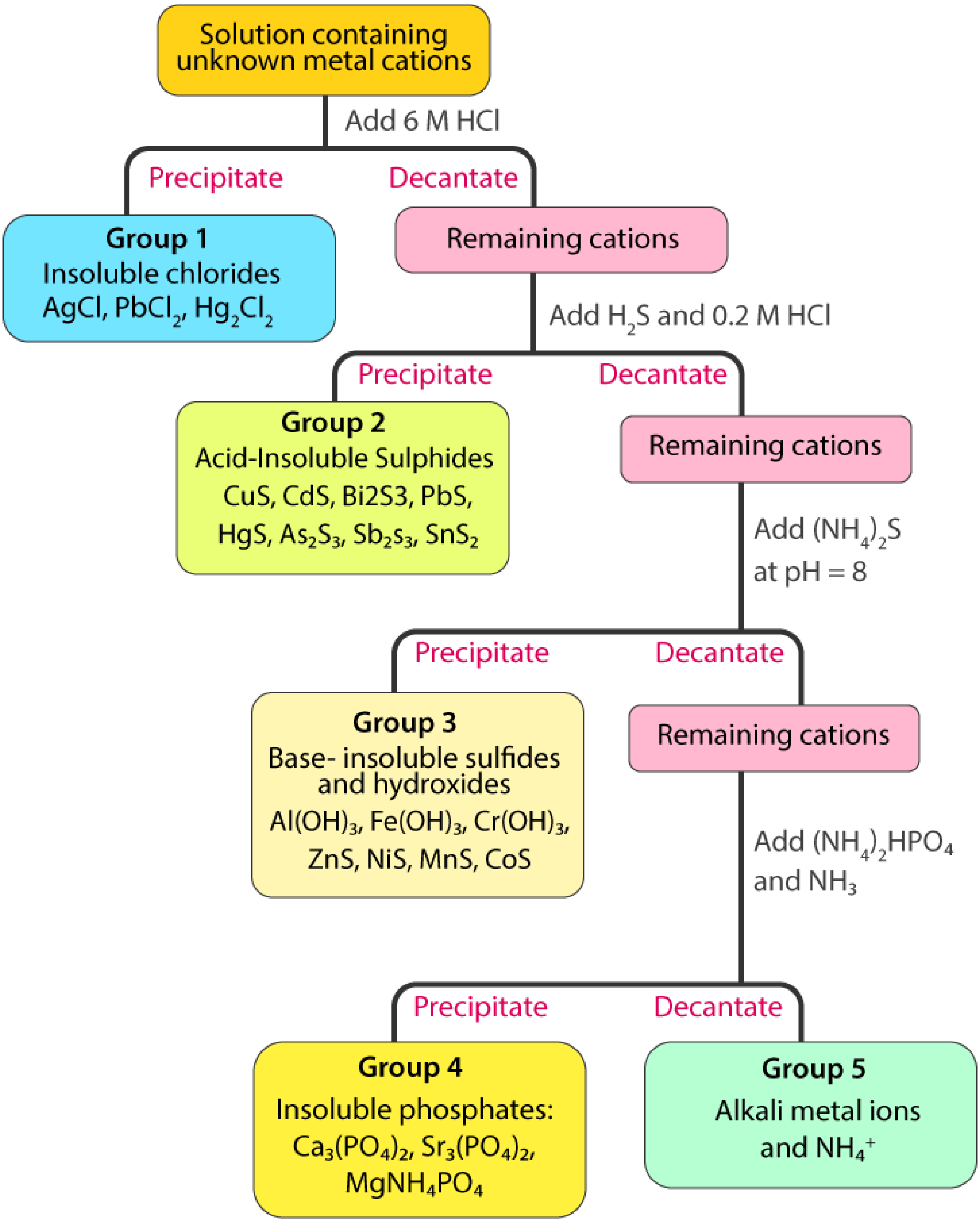
Hydrogen ions acts as common ion among them and higher concentration of H^+ suppresses the ionization of interfering acid. Therefore, ionic product of $C_2O_4^{2-}$ and Ba^{2+} doesn't exceed the solubility product of barium oxalate which is why Ba^{2+} remains in the solution as barium oxalate. That's how interfering radicals do not interfere as long as the medium remains acidic enough. But when we make the medium alkaline by adding 3rd group reagent ammonium hydroxide NH_4OH, OH^- ions combine with H^+ and neutralise them. This decreases the concentration of H^+ ions which shifts the equilibrium of dissociation of interfering acid forward and increases the concentration of $C_2O_4^{2-}$. Thus, the ionic product of $C_2O_4^{2-}$ and Ba^{2+} exceeds the solubility product of barium oxalate and Ba^{2+} gets precipitated in the 3rd group, which actually belongs to the 4th group.

Thus, the removal of interfering radicals is necessary to proceed the group III analysis.

Laboratory Techniques for Semi-micro–Qualitative Analysis:

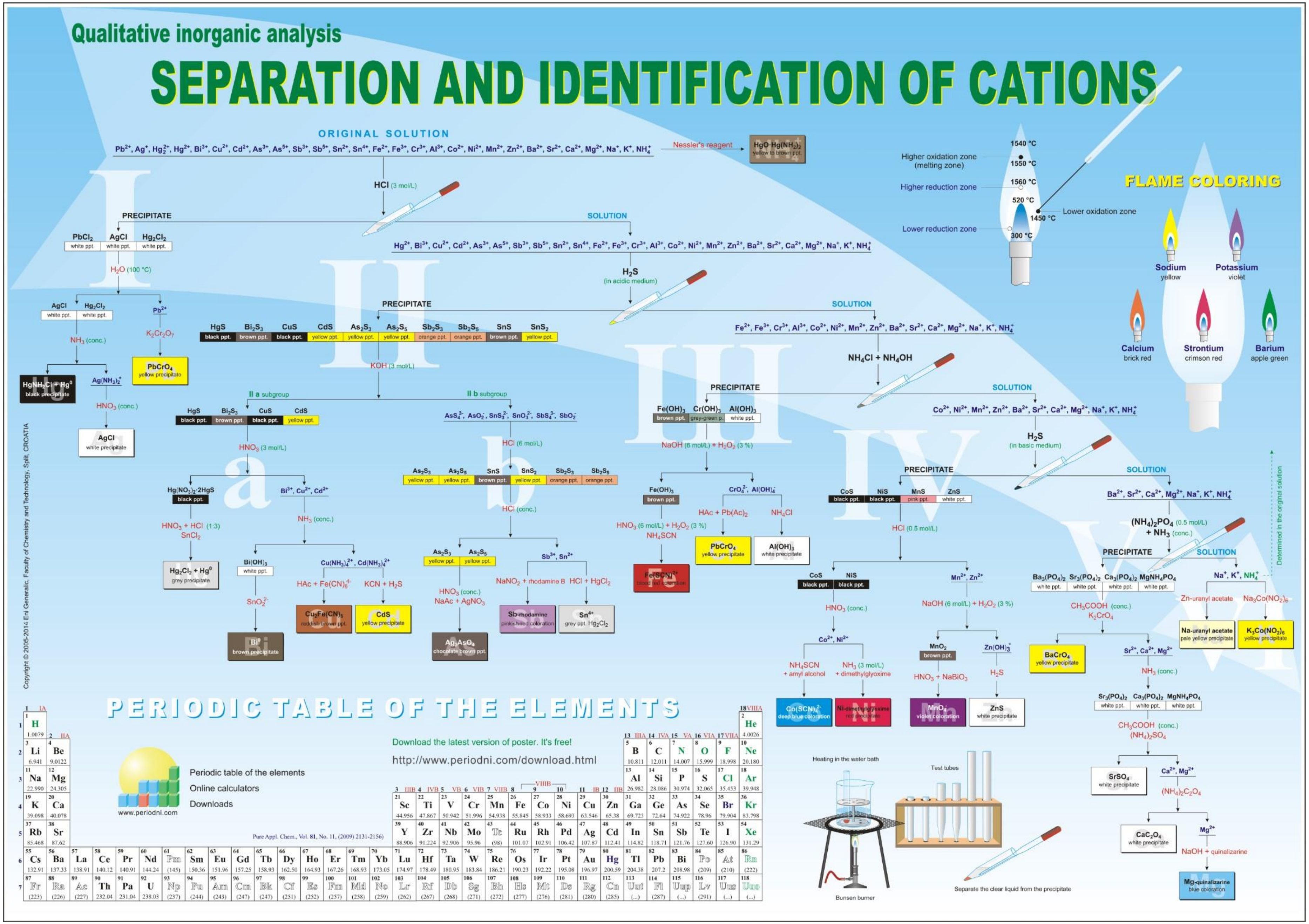
1) Distilled water is used at all times, never use tap water for any experiments.
2) It is essential that all glassware be clean, but not necessarily dry, before use.
3) Care should be taken to label test tubes and beakers in order to avoid confusion.
4) Do not place the reagent dropper tips into the mouth of the test tube. Hold them a little above the mouth to avoid contamination.
5) Estimate volumes (20 drops = 1 mL) whenever possible to save time.
6) Always mix solutions thoroughly by flicking the test tube. Never shake with your finger over the end.
7) The approximate pH of a solution is determined by removing a drop of the solution with a stirring rod and touching it to a piece of universal indicator paper.
8) A 100or 150 mL beaker of boiling distilled water is used to heat solutions in test tubes.
9) To perform a precipitation, add the indicated reagent to your test tube and mix thoroughly. Heat the solution if so directed (this sometimes help to form larger particles). When the precipitation appears to be complete, centrifuge the sample. Before removing the supernatant liquid, add a drop of the precipitating reagent. If the precipitation is incomplete, more precipitate will form in the liquid.
   If this is the case, add a few more drops, centrifuge, and test again.
10) Keep careful records of colors, textures, odors, reactions, etc in your observation notebook.

Salt Analysis Tips:



References:

1. Basic Principles of Practical Chemistry; Authors: Kulandaivelu A.R., Veeraswamy R., Venkateswaran V. Publisher: Sultan Chand & Sons; Publishing Year: 2017.


Qualitative inorganic analysis
SEPARATION AND IDENTIFICATION OF CATIONS
ORIGINAL SOLUTION
Pb²⁺, Ag⁺, Hg₂²⁺, Hg²⁺, Bi³⁺, Cu²⁺, Cd²⁺, As³⁺, As⁵⁺, Sb³⁺, Sb⁵⁺, Sn²⁺, Sn⁴⁺, Fe²⁺, Fe³⁺, Cr³⁺, Al³⁺, Co²⁺, Ni²⁺, Mn²⁺, Zn²⁺, Ba²⁺, Sr²⁺, Ca²⁺, Mg²⁺, Na⁺, K⁺, NH₄⁺
Nessler's reagent
HgO·Hg(NH₂)I yellow to brown ppt.
I
HCl (3 mol/L)
PRECIPITATE
PbCl₂ white ppt.
AgCl white ppt.
Hg₂Cl₂ white ppt.
H₂O (100 °C)
AgCl white ppt.
Hg₂Cl₂ white ppt.
Pb²⁺
NH₃ (conc.)
K₂Cr₂O₇
PbCrO₄ yellow precipitate
HgNH₂Cl + Hg⁰ black precipitate
Ag(NH₃)₂⁺
HNO₃ (conc.)
AgCl white precipitate
SOLUTION
Hg²⁺, Bi³⁺, Cu²⁺, Cd²⁺, As³⁺, As⁵⁺, Sb³⁺, Sb⁵⁺, Sn²⁺, Sn⁴⁺, Fe²⁺, Fe³⁺, Cr³⁺, Al³⁺, Co²⁺, Ni²⁺, Mn²⁺, Zn²⁺, Ba²⁺, Sr²⁺, Ca²⁺, Mg²⁺, Na⁺, K⁺, NH₄⁺
II
H₂S (in acidic medium)
PRECIPITATE
HgS black ppt.
Bi₂S₃ brown ppt.
CuS black ppt.
CdS yellow ppt.
As₂S₃ yellow ppt.
As₂S₅ yellow ppt.
Sb₂S₃ orange ppt.
Sb₂S₅ orange ppt.
SnS brown ppt.
SnS₂ yellow ppt.
KOH (3 mol/L)
II a subgroup
HgS black ppt.
Bi₂S₃ brown ppt.
CuS black ppt.
CdS yellow ppt.
a
HNO₃ (3 mol/L)
Hg(NO₃)₂·2HgS black ppt.
HNO₃ + HCl (1:3)
SnCl₂
Hg₂Cl₂ + Hg⁰ grey precipitate
Bi³⁺, Cu²⁺, Cd²⁺
NH₃ (conc.)
Bi(OH)₃ white ppt.
SnO₂²⁻
Bi⁰ brown precipitate
Cu(NH₃)₄²⁺, Cd(NH₃)₄²⁺
HAc + Fe(CN)₆⁴⁻
Cu₂Fe(CN)₆ reddish brown ppt.
KCN + H₂S
CdS yellow precipitate
II b subgroup
AsS₄³⁻, AsO₂⁻, SnS₃²⁻, SnO₃²⁻, SbS₄³⁻, SbO₂⁻
b
HCl (6 mol/L)
As₂S₃ yellow ppt.
As₂S₅ yellow ppt.
SnS brown ppt.
SnS₂ yellow ppt.
Sb₂S₃ orange ppt.
Sb₂S₅ orange ppt.
HCl (conc.)
As₂S₃ yellow ppt.
As₂S₅ yellow ppt.
HNO₃ (conc.)
NaAc + AgNO₃
Ag₃AsO₄ chocolate brown ppt.
Sb³⁺, Sn²⁺
NaNO₂ + rhodamine B
Sb-rhodamine pinkish-red coloration
HCl + HgCl₂
Sn⁴⁺ grey ppt. Hg₂Cl₂
SOLUTION
Fe²⁺, Fe³⁺, Cr³⁺, Al³⁺, Co²⁺, Ni²⁺, Mn²⁺, Zn²⁺, Ba²⁺, Sr²⁺, Ca²⁺, Mg²⁺, Na⁺, K⁺, NH₄⁺
III
NH₄Cl + NH₄OH
PRECIPITATE
Fe(OH)₃ brown ppt.
Cr(OH)₃ grey-green p.
Al(OH)₃ white ppt.
NaOH (6 mol/L) + H₂O₂ (3 %)
Fe(OH)₃ brown ppt.
HNO₃ (6 mol/L) + H₂O₂ (3 %)
NH₄SCN
Fe(SCN)²⁺ blood red coloration
CrO₄²⁻, Al(OH)₄⁻
HAc + Pb(Ac)₂
PbCrO₄ yellow precipitate
NH₄Cl
Al(OH)₃ white precipitate
SOLUTION
Co²⁺, Ni²⁺, Mn²⁺, Zn²⁺, Ba²⁺, Sr²⁺, Ca²⁺, Mg²⁺, Na⁺, K⁺, NH₄⁺
IV
H₂S (in basic medium)
PRECIPITATE
CoS black ppt.
NiS black ppt.
MnS pink ppt.
ZnS white ppt.
HCl (0.5 mol/L)
CoS black ppt.
NiS black ppt.
HNO₃ (conc.)
Co²⁺, Ni²⁺
NH₄SCN + amyl alcohol
Co(SCN)₄²⁻ deep blue coloration
NH₃ (3 mol/L) + dimethylglyoxime
Ni-dimethylglyoxime red precipitate
Mn²⁺, Zn²⁺
NaOH (6 mol/L) + H₂O₂ (3 %)
MnO₂ brown ppt.
HNO₃ + NaBiO₃
MnO₄⁻ violet coloration
Zn(OH)₄²⁻
H₂S
ZnS white precipitate
SOLUTION
Ba²⁺, Sr²⁺, Ca²⁺, Mg²⁺, Na⁺, K⁺, NH₄⁺
V
(NH₄)₂PO₄ (0.5 mol/L) + NH₃ (conc.)
PRECIPITATE
Ba₃(PO₄)₂ white ppt.
Sr₃(PO₄)₂ white ppt.
Ca₃(PO₄)₂ white ppt.
MgNH₄PO₄ white ppt.
CH₃COOH (conc.)
K₂CrO₄
BaCrO₄ yellow precipitate
Sr²⁺, Ca²⁺, Mg²⁺
NH₃ (conc.)
Sr₃(PO₄)₂ white ppt.
Ca₃(PO₄)₂ white ppt.
MgNH₄PO₄ white ppt.
CH₃COOH (conc.)
(NH₄)₂SO₄
SrSO₄ white precipitate
Ca²⁺, Mg²⁺
(NH₄)₂C₂O₄
CaC₂O₄ white precipitate
Mg²⁺
NaOH + quinalizarine
Mg-quinalizarine blue coloration
SOLUTION
VI
Na⁺, K⁺, NH₄⁺
Zn-uranyl acetate
Na-uranyl acetate pale yellow precipitate
Na₃Co(NO₂)₆
K₃Co(NO₂)₆ yellow precipitate
Determined in the original solution
Higher oxidation zone (melting zone)
Higher reduction zone
Lower reduction zone
Lower oxidation zone
1540 °C
1550 °C
1560 °C
520 °C
1450 °C
300 °C
FLAME COLORING
Sodium yellow
Potassium violet
Calcium brick red
Strontium crimson red
Barium apple green
Heating in the water bath
Test tubes
Bunsen burner
Separate the clear liquid from the precipitate
PERIODIC TABLE OF THE ELEMENTS
Download the latest version of poster. It's free!
http://www.periodni.com/download.html
Periodic table of the elements
Online calculators
Downloads
www.periodni.com
Pure Appl. Chem., Vol. 81, No. 11, (2009) 2131-2156)
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INORGANIC PREPARATIONS

1. Preparation of Tetraammine copper (II) sulphate

Aim

To prepare a pure sample of Tetraammonium Copper (II) Sulphate Tetrahydrate

Principle

When a solution of copper sulphate is mixed with an excess of ammonia solution, initially a white precipitate of cuprous hydroxide formed, dissolved again to give a deep blue solution of cuprammonium sulphate complex. The complex is reprecipitated from the solution by the addition of alcohol in which it is insoluble.

$$CuSO_4 + NH_4OH \rightarrow [Cu(NH_3)_4]SO_4.4H_2O$$

Chemicals Required

1.	Copper (II) sulphate	1.5 g
2.	Liquor ammonia	5 mL
3.	Ethanol	5 mL

Procedure

In a 100 mL beaker, **1.5 g of Copper Sulphate** is dissolved in about 10 mL of water. A few drops of **Conc. H_2SO_4** is added to it. To this solution, 1:1 ammonia (NH_3) is added slowly until clear blue liquid is obtained. If there appears a white turbidity, a little more ammonia (NH_3) is added. Now, 10mL alcohol is added drop by drop with constant stirring when a purple-coloured precipitate of Tetraammine copper (II) sulphate is obtained. The beaker is heated on a water bath to about 50°C for 10 – 15 minutes to dissolve the precipitate. The solution is cooled and the blue crystals of the complex are collected by filtering through funnel. The crystals are washed and dried

Result:

The amount of tetraammine copper (II) sulphate tetrahydrate _____________ g.

2. Preparation of hexa thiourea lead (II) nitrate

Aim

To prepare hexa thiourea lead (II) nitrate, other names - hexathiourea plumbus (II) nitrate.

Principle

When aqueous solution of Lead nitrate is heated with aqueous solution of Thiourea, white crystals are obtained. The reaction involved in the formation of white crystals of hexa thiourea lead (II) nitrate.

$$\mathbf{Pb\ (NO_3)_2 + 6\ NH_2CSNH_2 \rightarrow [Pb\ (NH_2CSNH_2)]_6\ (NO_3)_2}$$

Chemicals Required

1. Lead nitrate [Pb $(NO_3)_2$]	500 mg
2. Thiourea (NH_2CSNH_2)	650 mg
3. Distilled water.	5 - 10 mL

Procedure

Dissolve 500 mg of lead nitrate in 5 - 10 mL of distilled water in beaker then heat it. Dissolve 650 mg of thiourea in 5 - 10 mL of distilled water. Mix the two solutions in one beaker and then cool the mix with ice bath; this will lead to produce separated colourless crystals in the form of needles. Filter the solution, dry the crystals and weight out them.

Result:

The amount of hexa thiourea lead (II) nitrate ______________ g.

3. Preparation of Potassium trioxalato chromate (III)

Aim

To prepare a pure complex of the crystals of Potassium trioxalato chromate (III), $K_3[(Cr(C_2O_4)_3].3H_2O$

Principle

When potassium dichromate is treated with oxalic acid and potassium oxalate, a green complex of potassium trioxalato chromate (III) is formed.

$$2K_2C_2O_4 + 7C_2H_2O_4 + K_2Cr_2O_7 \longrightarrow K_3[(Cr(C_2O_4)_3].3H_2O$$

Chemicals Required

1. Potassium oxalate ($K_2C_2O_4$)	1.5 g
2. Oxalic acid ($H_2C_2O_4$. $2H_2O$)	3.5 g
3. Potassium Dichromate ($K_2Cr_2O_7$)	1.3 g
4. Acetone	5 mL

Procedure

1.5 g of Potassium oxalate and 3.5 g Oxalic acid are dissolved in 50 mL of water taken in a 100 mL conical flask. Potassium dichromate crystals are added in small quantities at a time to the solution and the solution is vigorously stirred. When the reaction is complete, the solution is evaporated to nearly one third of its original volume and allowed to stand over night when green coloured crystals are obtained. The crystals are filtered at the pump, washed with acetone and dried. The yield is recorded.

Result

The weight of Potassium trioxalato chromate (III) obtained _____________ g.

4. Preparation of Tristhiourea copper (I) Sulphate

Aim

To prepare a pure sample of tris-(thiourea)-Copper (I) Sulphate dihydrate

$\left[Cu(NH_2\text{-}CS\text{-}NH_2)_3\right]_2 SO_4.2H_2O$

Principle

When a solution of copper sulphate and thiourea are shaken together they readily yield the complex. In the first step, thiourea reduces Cu^{+2} to Cu^{+1} and then forms the complex as follows.

$$\begin{array}{l} CuSO_4 + NH_2\text{-}CS\text{-}NH_2 \longrightarrow Cu_2SO_4 + NH_2\text{-}CS\text{-}NH\text{-}HSO_4 \\ Cu_2SO_4 + 6NH_2\text{-}CS\text{-}NH_2 \longrightarrow \left[Cu(NH_2\text{-}CS\text{-}NH_2)_3\right]_2 SO_4.2H_2O \\ \hline 7CuSO_4 + 7NH_2\text{-}CS\text{-}NH_2 \longrightarrow \left[Cu(NH_2\text{-}CS\text{-}NH_2)_3\right]_2 SO_4.2H_2O + NH_2\text{-}CS\text{-}NH\text{-}HSO_4 \end{array}$$

White Crystalline precipitate

Chemicals required

1. Copper Sulphate (**$CuSO_4.5H_2O$**)	1g
2. Thiourea, (**$NH_2\text{-}CS\text{-}NH_2$**)	1g

Procedure

1 g of thiourea crystals are dissolved in 5-10 mL of hot water in a round bottom flask and cooled the flask to room temperature. A solution of copper sulphate is prepared by dissolving 1 g of it in 5-10 mL of water. The flask is stoppered well and thoroughly shaken after each addition. When the addition copper sulphate solution is completed, the flask is again shaken well for another ten minutes. At this stage, tris complex will begin to appear. The contents of the R, B flask are heated on a water bath to about 70 °C - 80 °C for 15 – 20 minutes. The flask is cooled to room temperature under tap water.

The mother liquor is decanted off. The oily liquid or they crystals formed at the sides of the flask are thoroughly shaken with 10 % solution of thiourea. Well defined white crystals are formed. The crystals are filtered at the pump, washed with distilled water and dried in a desiccator. The yield is recorded.

Result

The weight of Tristhiourea copper (I) Sulphate dihydrate__________ g.

5. Preparation of Hexammine cobalt (III) chloride

Aim

To prepare Hexammine cobalt (III) chloride involves the mixing of ammonium chloride and cobaltous chloride followed by the addition of conc. Ammonia and hydrogen peroxide.

Principle

Preparation of Hexammine Cobalt (III) Chloride involves the mixing of ammonium chloride and cobaltous chloride followed by the addition of conc. Ammonia and hydrogen peroxide.

$$2CoCl_2 + 2NH_4Cl + IONH_3 + H_2O_2 \longrightarrow 2[Co(NH_3)_6]Cl_3 + 2H_2O$$

Chemicals Required

1. Ammonium Chloride (NH_4Cl)	1 g
2. Cobaltous chloride ($CoCl_2$)	2 g
3. 20 Volume Hydrogen Peroxide (H_2O_2)	5 - 10 mL

Procedure

2g of Cobaltous chloride is dissolved in a boiling solution of 1g of Ammonium Chloride in 15 mL of water. Add 1 g of animal charcoal and cool the contents in the running water. Wash out the vessel in which the solution was first made with 40 mL of Conc. NH_3 and then add the ammoniacal liquid to the flask. Cool the whole contents of precipitation in ice bath to 10 °C. 5-10 mL of 20 Volume Hydrogen Peroxide is added slowly in portions while briskly shaking the flask and its contents. All the oxidising having been added, heat the mixture gradually to 60 °C with mixing by shaking until the pinkish tint in the liquid is removed. Crystals begin to separate at the close of heating and deposit in quantity on cooling in an ice bath.

Filter off the crude solid and without washing it, transfer it to a beaker containing a boiling mixture of 25 mL of water and 2 mL of conc. HCl. When all solid except the charcoal has dissolved, filter the liquid while hot. Add 5 mL of conc. HCl to the filtrate and then cool the cool the liquid in an ice bath. Golden brown crystals separate out.

Result:

The amount of hexammine cobalt (III) Chloride ______________ g.

6. Preparation of Iron (III) hexacyanoferrate (II) or Prussian Blue $Fe_4[Fe(CN)_6]_3$

Aim

To prepare a pure sample of crystals of Prussian blue, $Fe_4[Fe(CN)_6]_3$

Principle

Prussian blue is prepared by mixing ferric chloride ($FeCl_3$) Solution with potassium ferrocyanide solution, $K_4[Fe(CN)_6]$.

$$4FeCl_3 + 3K_4[Fe\,(CN)_6] \rightarrow Fe_4[Fe(CN)_6]_3 + 12\,KCl$$

Chemicals required

1. Ferric Chloride **(**$FeCl_3$)	1 g
2. Potassium Ferrocyanide $K_4[Fe\,(CN)_6]$	2.5 g

Procedure

1 g Ferric chloride and 2.5 g Potassium Ferrocyanide are separately dissolved in 5 - 10 mL of distilled water in 100 mL of beakers. Then, these two solutions are mixed in a 250 mL beaker. If necessary, the solution may be heated on a wire-gauze for half an hour and cooled slowly. A blue colour crystal separated from the solution. The crystals are filtered and dried with the help of filter papers.

Result

The amount of Prussian blue obtained _______________ g.