

**SEMI MICRO QUALITATIVE ANALYSIS
OF
INORGANIC CATION MIXTURE**

For use by students of **M.Sc. I**, Chemistry, **Semester– I**

Government Institute of Science, Nagpur

**Affiliated
to
Rashtrasant Tukdoji Maharaj Nagpur University, Nagpur.**

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M.Sc.I ,Chemistry Practical Syllabus for Semesters 1

CH-105 INORGANIC CHEMISTRY – PRACTICALS-I

I - Preparation of Inorganic Complexes and their characterization by:

Elemental analysis and physico-chemical methods (Electronic and IR Spectra, magnetic susceptibility measurements, Thermal analysis and Molar conductance studies).

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|----------------------------------|------------------------|---------------------------------|
| 1. $K_3 [Al (C_2O_4)_3](H_2O)_3$ | 2. $[VO (acac)_2]$ | 3. $Na [Cr (NH_3)_2(SCN)_4]$ |
| 4. $K_3[Cr(SCN)_6]$. | 5. $[Mn (acac)_3]$ | 6. $K_3 [Fe (C_2O_4)_3]$ |
| 7. $Hg [Co (SCN)_4]$ | 8. $[Co (Py)_2 Cl_2]$ | 9. $[Cu_2 (CH_3COO)_4(H_2O)_2]$ |
| 10. $[Ni (DMG)_2]$ | 11. $[Ni(NH_3)_6]Cl_2$ | 12. $[Cu(NH_3)_4(H_2O)_2]SO_4$ |

II– Quantitative analysis

Separation and determination of two metal ions from the following alloys involving: Volumetric, Gravimetric and Spectrophotometric methods.

- Copper (II) and Nickel (II)
- Copper (II) and Zinc (II)
- Nickel (II)—Zinc (II) and
- Copper (II)—Iron (III)

III – Qualitative analysis

Semi-micro Analysis of inorganic mixture containing four cations out of which two will be rare metal ions such as W, Mo, Se, Ti, Zr, Ce, Th, V and U. (**Spot Test** for individual cations should be performed).

References

- Vogel – A Text Book of Qualitative Inorganic Analysis – Longman
- Kolthoff & Stenger – Volumetric Analysis – Interscience
- Vogel – A Text Book of Quantitative Inorganic Analysis – Longman
- Kolthoff & Sandell - Text Book of Qualitative Inorganic Analysis.
- G. Schwarzen Back “Complexometric Titration” Interscience.

Note to Examiners:

- Candidates may be asked to report four metal ions present in the given mixture.
- While reporting the scheme of analysis the student is expected to indicate the chemistry involved in the relevant reactions.
- The candidates may be asked to give the procedure for the quantitative analysis giving the chemistry behind the experiments.
- Each student has to carry out II (30 marks) and III (20 marks) experiments for the practical examination.

SCHEME FOR SEPARATION AND IDENTIFICATION OF INORGANIC CATIONS IN SOLUTION
For use by students of M. Sc. I Chemistry, Semesters -1, RashtaraSant Tukdoji Maharaj Nagpur University, Nagpur

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References: Vogel's Qualitative Inorganic Analysis 6th edn., revised by G.Svehla, Orient Longman, 1979

Ions to be identified:

- a) Ammonium (NH₄⁺), Silver (Ag⁺), Mercurous (Hg⁺), Mercuric (Hg²⁺), Lead (Pb²⁺), Bismuth (Bi³⁺), Cupric (Cu²⁺), Cadmium(Cd²⁺), Arsenic (As³⁺), Antimony (Sb³⁺), Stannous (Sn²⁺), Stannic (Sn⁴⁺), Aluminum (Al³⁺), Chromic (Cr³⁺), Ferrous (Fe²⁺), Ferric (Fe³⁺), Manganous (Mn²⁺), Cobalt (Co²⁺), Nickel (Ni²⁺), Zinc (Zn²⁺), Calcium (Ca²⁺), Strontium (Sr²⁺), Barium (Ba²⁺), Magnesium (Mg²⁺).
- b) Thallium (Tl⁺), Tungsten as tungstate (WO₄²⁻), Selenium as selenite (SeO₃²⁻) or selenate (SeO₄²⁻), Tellurium as tellurite (TeO₃²⁻) or tellurate (TeO₄²⁻), Molybdenum as molybdate (MoO₄²⁻), Cerium (Ce⁴⁺), Thorium (Th⁴⁺), Titanium (Ti⁴⁺), Zirconium as zirconyl (ZrO²⁺), Vanadium as metavanadate (VO₃²⁻), Uranium as uranyl (UO₂²⁺), Lithium (Li⁺).

Preparation of the Original solution: Take small quantity of the powdered solid and examine the solubility in the following solvents in the order given:

1. Water (Cold and hot)
2. Dil. HCl (Cold and hot)
3. Conc. HCl (Cold and hot)
4. Dil. HNO₃ (Cold and hot)
5. Conc. NHO₃ (Cold and hot)
6. Aqua Regia (3 parts of Conc. HCl + 1 part of Conc. HNO₃, mix and use the solution immediately, do not keep it at all)

The given aqueous solution shall contain four cations, two cations from (a) and two from (b).

CATION GROUPS AND REAGENTS

Group	Group Reagent	Cations (some metals as more stable oxy anions)
Group I	Dil. HCl (2M)	Hg ⁺ , Ag ⁺ , Pb ²⁺ , Tl ⁺ , WO ₄ ²⁻ .
Group II	Dil. HCl + H ₂ S gas	Hg ²⁺ , Pb ²⁺ , Bi ³⁺ , Cu ²⁺ , Cd ²⁺ . As ³⁺ , Sb ³⁺ , Sn ²⁺ , Sn ⁴⁺ , SeO ₃ ²⁻ , SeO ₄ ²⁻ , TeO ₃ ²⁻ , TeO ₄ ²⁻ , MoO ₄ ²⁻
Group III A	NH ₄ Cl + NH ₄ OH solutions (2M)	Al ³⁺ , Cr ³⁺ , Fe ²⁺ , Fe ³⁺ , Ce ⁴⁺ , Th ⁴⁺ , Ti ⁴⁺ , ZrO ²⁺ , VO ₃ ²⁻ , UO ₂ ²⁺ .
Group III B	NH ₄ Cl + NH ₄ OH+ H ₂ S gas	Co ²⁺ , Ni ²⁺ , Mn ²⁺ , Zn ²⁺ .
Group IV	NH ₄ Cl + NH ₄ OH + (NH ₄) ₂ CO ₃ solutions (2M)	Ca ²⁺ , Sr ²⁺ , Ba ²⁺ .
Group V	NH ₄ OH + (NH ₄) ₂ HPO ₄ or Na ₂ HPO ₄	Mg ²⁺
Other Radicals	No common reagent	NH ₄ ⁺ , Na ⁺ , Li ⁺ , K ⁺

Instructions:

1. Chemistry, and science in general, depends a great deal on the observational skill of its practitioners. Therefore cultivate the habit of recording your observations honestly and in detail, irrespective of what may be given in the scheme (in spite of all efforts, the scheme may not be totally devoid of errors). This may save you time in case you make wrong conclusions or, hopefully, provide new information valuable to science. It will also help improve your own knowledge of the subject.
2. Maintain your observation books and fair records neatly, with appropriately numbered 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.
3. Maintain strict discipline inside the laboratory and concentrate on your work. Discipline is necessary for safety, and helps reduce breakage. Remember, your performance is being continuously assessed.
4. Write down the procedure for the test and decide what to do before doing it. Record your observations as soon as they are made while the impressions are still fresh in your memory, and not later. Nobody has a perfect memory. Marks are awarded by the examiners based on what you have recorded and not on what you thought. Whenever you come for discussions, clarifications or reporting results, recorded observations up to that point **must** be presented.
5. All tests should be done systematically in the given order since the order in which the tests are done is very important.
6. 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.
7. 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.
8. 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.
9. 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.
10. 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.
11. Using 1 cm³ of the given mixture, carry out the group tests alone to identify which groups are present. Only the reagents for groups which are present need 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 cm³ of the mixture should be sufficient for complete analysis.
12. 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.
13. **Use only distilled water for dilution and washing in all tests**

Tests for ammonium, (NH₄)⁺*Note: Do the tests for ammonium using the original solution given.*

Test	Observation	Inference
1. Add 0.5 cm ³ of NaOH solution (2M) to 0.5 cm ³ of the given solution in a test tube and heat gently to boiling. Smell the mouth of the tube occasionally while heating.	Characteristic smell of ammonia	Ammonium may be present. Strong alkalis liberate ammonia from ammonium salts. $\text{NH}_4^+ + \text{OH}^- \longrightarrow \text{NH}_3 \uparrow + \text{H}_2\text{O}$
2. Add 0.5 cm ³ of NaOH solution (2M) to 0.5 cm ³ of the given solution in a test tube and heat gently to boiling. Insert a glass rod dipped in conc. HCl into the mouth of the test tube while heating.	Copious white fumes from the glass rod.	Ammonium may be present. The liberated ammonia reacts with HCl producing fumes of NH ₄ Cl.
3. To 0.5 cm ³ of saturated sodium hydrogen tartrate solution (or saturated tartaric acid solution) in a test tube, add a few drops of the test solution.	A white crystalline precipitate is formed.	Presence of NH ₄ ⁺ is confirmed. The precipitate is ammonium hydrogen tartrate. $\text{H}_2\text{C}_4\text{H}_4\text{O}_6 + \text{NH}_4^+ \longrightarrow \text{NH}_4\text{HC}_4\text{H}_4\text{O}_6 \downarrow + \text{H}^+$
4. To 0.5 cm ³ of saturated sodium hexanitrito cobaltate (III) solution, add a few drops of the test solution.	A yellow precipitate is formed.	Presence of NH ₄ ⁺ is confirmed. The precipitate is ammonium hexanitritocobaltate(III). $3 \text{NH}_4^+ + [\text{Co}(\text{NO}_2)_6]^{3-} \longrightarrow (\text{NH}_4)_3[\text{Co}(\text{NO}_2)_6] \downarrow$
Spot test: Mix a drop of the test solution with a drop of sodium hydroxide solution on a watch glass. Place a micro drop of this mixture on a piece of filter paper and add a drop of Nessler's reagent on it.	An orange red stain or ring is produced.	Presence of NH ₄ ⁺ is confirmed. The colour is due to the formation of ammonium mercuric iodide. <i>(Note: Various formulae have been reported for the product by different authors).</i>

Separation of Cations into Groups.

Note: After centrifuging in each group, transfer centrifugate into a small beaker for evaporation. Carry out precipitation in the beaker itself and then transfer into the centrifuge tube.

Add a few drops of dilute (2M) HCl to 5 cm ³ of the cold test solution. If a precipitate forms, continue adding the acid in drops with shaking till no further precipitation takes place. Centrifuge.							
<p>Residue: Chlorides of Group I metals Or H₂WO₄.H₂O.</p> <p>White - Hg⁺, Ag⁺, Pb²⁺, Tl⁺, or WO₄²⁻.</p> <p>Examine as given in Table 1.</p>	<p>Centrifugate: Add a drop of H₂O₂ and dilute the solution to double its volume with water. Heat nearly to boiling and saturate with H₂S gas. Shake well. If a precipitate forms, continue passing H₂S till no more precipitate is formed and smell of H₂S persists. Centrifuge.</p>						
	<p>Residue: Sulphides of Group II metals.</p> <p>Black – Hg²⁺, Pb²⁺, Bi³⁺ or Cu²⁺. Yellow – Cd²⁺, Sn⁴⁺ Se or As³⁺. Orange red - Sb³⁺. Brown -Te, Sn²⁺ or Mo⁶⁺.</p> <p>Examine as given in Table 2.</p>	<p>Residue: Hydroxides or oxides of Group III A metals.</p> <p>White - Al³⁺, VO₃²⁻, Ti⁴⁺, ZrO²⁺ or Th⁴⁺; White, Slowly turning Brown -Ce⁴⁺ Bluish green - Cr³⁺; Brown - Fe³⁺; Yellow - UO₂²⁺.</p> <p>Examine as given in Table 3.</p>		<p>Centrifugate: Boil down to half its volume in a small beaker till no smell of H₂S remains. Add 1 cm³ of concentrated HNO₃ (to oxidize any Fe²⁺ to Fe³⁺). Evaporate till almost dry and dilute to 5 cm³ with water. Add 2 g of solid NH₄Cl and heat to boiling. Add dilute NH₃ solution drop by drop with shaking till a strong smell of ammonia persists. Heat to boiling and centrifuge.</p>			
		<p>Sulphides of Group III B metals.</p> <p>Black - Co²⁺ or Ni²⁺; Pink - Mn²⁺; White - Zn²⁺.</p> <p>Examine as given in Table 4.</p>	<p>Centrifugate: Evaporate to reduce volume. Add dilute NH₃ solution drop by drop with shaking till a strong smell of ammonia persists. Saturate with H₂S gas. Shake well. If a precipitate forms, continue passing H₂S till no more precipitate is formed and smell of H₂S persists. Boil. Centrifuge.</p>		<p>Centrifugate: Evaporate till almost dry and dilute to 5 cm³ with water. Add dilute NH₃ solution drop by drop with shaking till a strong smell of ammonia persists. Add (NH₄)₂CO₃ solution dropwise with stirring till precipitation is complete.</p>		
			<p>Carbonates of Group IV metals.</p> <p>White - Ca²⁺, Sr²⁺ or Ba²⁺.</p> <p>Examine as given in Table 5.</p>	<p>Residue: Carbonates of Group IV metals.</p>		<p>Centrifugate: Boil and reduce to half volume of solution then make it alkaline with NH₄OH, +4-5 drops of 5 % (NH₄)₂HPO₄ or Na₂HPO₄, boil and allow it to cool and centrifuge.</p>	
				<p>Residue : :(Group V Alkaline) White – MgNH₄PO₄ May contain Mg²⁺ Examine as given in Table 6.</p>		<p>Centrifugate : :(Group Other Radical) Use W.E for the test of K⁺, Na⁺, Li⁺ and NH₄⁺ Examine as given in Table 7.</p>	

Table 1.

<p>The Group I (Silver Group) residue is washed using 2 cm³ of dilute HCl (see instruction 9) and then with 2 cm³ of water. Discard the washings. Boil residue in the centrifuge tube with 2 cm³ of water and centrifuge while hot. Decant the centrifugate into a test tube. Repeat extraction with 2 cm³ boiling water and combine the centrifugate.</p>			
<p>Residue: White – Hg₂Cl₂ or AgCl. Yellow - H₂WO₄. Boil the residue with 5 cm³ of dilute NH₃ solution and centrifuge.</p>		<p>Centrifugate: May contain Pb²⁺ or Tl⁺, which may crystallize on cooling. Carefully add 1 cm³ of conc. H₂SO₄ and evaporate till fumes are seen. Cool and dilute carefully to 5 cm³ with water. If a precipitate is formed, centrifuge.</p>	
<p>Residue: Black precipitate, consisting of a mixture of Hg(NH₂)Cl and Hg.</p> <p>Dissolve the residue by heating with 2 cm³ of aqua regia, cool and do the following tests:</p> <p>1) To 1 cm³ of the solution, add NaOH solution in drops with shaking till excess. A black precipitate of Hg₂O is formed.</p> <p>2) To 1 cm³ of the solution, add K₂CrO₄ solution and heat. A brown precipitate changing to red Hg₂CrO₄ on heating.</p> <p>3) Spot test: Place a drop of faintly acid test solution (original) on a drop reaction paper and add a drop of saturated potassium nitrite (KNO₂) solution. A black spot of Hg is produced.</p> <p style="text-align: center;">Presence of Hg(I) is confirmed.</p>	<p>Centrifugate: May contain [Ag(NH₃)₂]⁺ or H₂WO₄. Add dilute HCl in drops with shaking till a faint precipitate forms, which is re-dissolved by adding one drop of dilute NH₃ solution. Add KI solution drop wise with shaking till precipitation, if any, is complete. Centrifuge.</p>	<p>Residue: White – PbSO₄. Dissolve in ammonium acetate solution (or a mixture of NH₃ solution and acetic acid) and the following tests are done:</p> <p>1) To 1 cm³ of the solution, add acetic acid and K₂CrO₄ solution. A yellow precipitate of PbCrO₄ is formed.</p> <p>2) To 1 cm³ of the solution, add KI solution. Boil and cool. A yellow precipitate of PbI₂ is formed, which dissolves on heating and re-precipitates as golden spangles on cooling.</p> <p>3) Spot test: Place a drop of the solution on a drop reaction paper and add a drop of ammonium sulphide solution. A black spot of PbS is formed.</p> <p style="text-align: center;">Presence of Pb (II) is confirmed.</p>	<p>Centrifugate: May contain Tl⁺. Add dilute NH₃ solution and boil off excess NH₃. Cool and do the following tests:</p> <p>1) To 1 cm³ of the solution, add acetic acid and K₂CrO₄ solution. A yellow precipitate of Tl₂CrO₄ is formed.</p> <p>2) Flame test: Evaporate 2 cm³ of the solution to dryness in a china dish, cool and mix residue with a drop of conc.HCl using a glass rod. A speck of the mixture is taken at the tip of a nickel spatula and introduced into a Bunsen flame. An intense green colour appears</p> <p>3) Spot test: Place a drop of the solution on a drop reaction paper and add a drop sodium thiosulphate solution. Add a drop of KI solution over it. A yellow spot of Tl (I) is produced.</p> <p style="text-align: center;">Presence of Tl (I) is confirmed.</p>
	<p>Residue: Pale yellow precipitate of AgI.</p> <p>1) To a portion of the above precipitate, add excess of sod. thiosulphate solution. The precipitate dissolves. Presence of Ag⁺ confirmed.</p> <p>2) Spot test: Place a drop of the test solution (original) on a watch glass and add a drop of ammonium carbonate solution and mix. Withdraw a drop of clear liquid from the mixture and place it on a drop reaction paper and add a drop of K₂CrO₄ solution. A red ring is obtained due to formation of Ag₂CrO₄.</p> <p style="text-align: center;">Presence of Ag(I) is confirmed.</p>		

Table 2.

<p>Wash the Group II residue with 2 cm³ of a solution of ammonium chloride saturated with H₂S and centrifuge. Discard centrifugate. Transfer residue into a small beaker and add 5 cm³ of yellow ammonium sulphide (amm. polysulphide) solution. Heat to about 50 to 60⁰C and maintain at this temperature for 5 minutes with stirring. If there is a precipitate, cool and centrifuge.</p>	
<p>Residue: Black – HgS, PbS, Bi₂S₃ or CuS. Yellow – CdS.</p> <p style="text-align: center;">Analyse as given in Table 2A.</p>	<p>Centrifugate: Add conc. HCl dropwise till just acidic (do not add excess!) and warm while stirring. A fine white or pale yellow precipitate is only sulfur. If a flocculant precipitate is formed, centrifuge. If not, discard the solution.</p>
	<p>Residue: Yellow – SnS₂, Se or As₂S₃. Orange red – Sb₂S₃. Brown -Te, SnS or MoS₃.</p> <p style="text-align: center;">Analyse as given in Table 2B.</p>

Table 2A

The Group IIA (Copper Group) residue is washed two times using 2 cm ³ of distilled water and centrifuged. Discard washings. Transfer residue to a small beaker, add 10 cm ³ of dilute nitric acid and boil for 5 minutes. If some solid material remains, cool and centrifuge.			
Residue: Black – HgS. Dissolve by boiling with 2 cm ³ of aqua regia. Do the following tests with this solution: 1) To 0.5 cm ³ of the solution, add NaOH solution dropwise with shaking till excess. A brownish red precipitate changing to yellow HgO. 2) To 0.5 cm ³ of the solution, add Na ₂ CO ₃ solution dropwise and shake till effervescence just stops. Then add KI solution dropwise with shaking till excess. A red precipitate of HgI ₂ is formed, which dissolves in excess reagent. 3) Spot test: Place a drop of the test solution on a spot plate. Add a drop of amm.thiocyanate solution and a drop of cobalt acetate solution. A blue colour is produced. <p style="text-align: center;">Presence of Hg(II) is confirmed.</p>	Centrifugate: May contain nitrate of Pb, Bi, Cu or Cd. Add dilute H ₂ SO ₄ till precipitation (if any) is complete. Centrifuge.		
	Residue: White – PbSO ₄ . Dissolve in ammonium acetate solution (or a mixture of NH ₃ solution and acetic acid) and the following tests are done: 1) To 1 cm ³ of the solution, add acetic acid and K ₂ CrO ₄ solution. A yellow precipitate of PbCrO ₄ is formed. 2) To 1 cm ³ of the solution, add KI solution. Boil and cool. A yellow precipitate of PbI ₂ is formed, which dissolves on heating and re-precipitates as golden spangles on cooling. 3) Spot test: Place a drop of the solution on a drop reaction paper and add a drop of amm. Sulphide solution. A black spot of PbS is formed. <p style="text-align: center;">Presence of Pb(II) is confirmed.</p>	Centrifugate: May contain nitrate or sulphate of Bi, Cu or Cd. Add concentrated NH ₃ solution dropwise with shaking till smell of ammonia persists. If a residue remains, centrifuge. Residue: White – Bi(OH) ₂ . The residue is divided into two parts. 1) To one part of the residue, add a few drops of conc. H ₂ O ₂ solution. A yellowish brown precipitate of bismuthate ions (BiO ₃ ⁻) is produced. 2) Dissolve the second part of the residue in 1 cm ³ of dilute HNO ₃ and divide into two parts. To one part, add KI solution dropwise with shaking to excess. A black precipitate of BiI ₃ is first formed, which dissolves in excess KI to give an orange solution of [BiI ₄ ⁻]. 3) Spot test: Place a drop of the second part on a spot plate and add a drop of thiourea solution. An intense yellow colour is produced. <p style="text-align: center;">Presence of Bi(III) is confirmed</p>	Centrifugate: May contain [Cu(NH ₃) ₄] ³⁺ or [Cd(NH ₃) ₄] ³⁺ . If deep blue in colour, Cu is present. Add dilute acetic acid with shaking till a clear solution is obtained. Divide into two parts. Divide the first part into three equal portions and do the following tests: 1) To one part, add pot. Ferrocyanide solution. A reddish brown precipitate of copper ferrocyanide is formed. 2) To the second portion, add pot.thiocyanate solution. A black precipitate turning slowly white is formed. $2 \text{ Cu(SCN)}_2 \rightarrow 2 \text{ CuSCN} + (\text{SCN})_2$ 3) Spot test: Place a drop of the third part on a spot plate and add a drop of tartaric acid solution, followed by a drop of NaOH solution. An intense blue colour is produced. <p style="text-align: center;">Presence of Cu(II) is confirmed</p>

Table 2B

Transfer the Group IIB (Tin Group) residue into a small conical flask and place a funnel in its mouth. Add 5 cm ³ of conc. HCl and boil gently for 5 minutes. Dilute with 3 cm ³ of distilled water, cool and centrifuge.			
Residue: Yellow - Se or As ₂ S ₃ . Brown -Te or MoS ₃ . Dissolve in 5 cm ³ of conc. HCl containing a pinch of solid potassium chlorate (KClO ₃). Transfer solution to a small beaker and concentrate by evaporating on a water bath. Cool and add NH ₃ solution with stirring till smell of ammonia persists. Add 2 cm ³ of Mg(NO ₃) ₂ solution and stir for 5 minutes. When precipitation is complete, centrifuge.		Centrifugate: May contain Sb or Sn as chloride. Boil to expel H ₂ S. Cool and carry out the following tests using small portions of the solution.	
Residue: White crystalline Mg(NH ₄)AsO ₄ .6H ₂ O Shake the residue well with 3cm ³ of water and divide into three portions. 1) To one portion, add AgNO ₃ solution containing a few drops of acetic acid. A red precipitate of silver arsenate, Ag ₃ AsO ₄ , is formed. 2) To the second portion, add 0.5 cm ³ of conc. HNO ₃ and excess of amm. molybdate solution. A yellow precipitate of amm. arsenomolybdate is formed. 3) Spot test: To the third portion, add two drops of conc. H ₂ O ₂ solution and warm. Acidify with a few drops of acetic acid. Place a drop of this mixture on a drop reaction paper followed by a drop of AgNO ₃ solution. A red spot is obtained. Presence of As is confirmed.	Centrifugate: May contain Se, Te or Mo as chlorides. Transfer to a small beaker and boil off NH ₃ . Add 2 cm ³ of conc. HCl and evaporate to half volume on a water bath. Add 2 cm ³ of a saturated solution of sodium sulphite (Na ₂ SO ₃) solution. If a precipitate is formed, centrifuge.		To 2 cm ³ of the solution in a test tube, add a pinch of iron filings and boil to reduce Sn(IV) to Sn(II). Cool and centrifuge. With draw the clear supernatant carry out the following tests. 1) To 1 cm ³ of the solution in a test tube, add two drops of mercuric chloride (HgCl ₂) solution. A silky white or grey precipitate of Hg ₂ Cl ₂ is obtained. 2) To 0.5 cm ³ of the solution in a test tube, add NaOH solution dropwise with shaking. A white precipitate of Sn(OH) ₂ soluble in excess NaOH is obtained. 3) Spot test: Place a drop of the solution on a drop reaction paper and add a drop of bismuth nitrate and a drop of NaOH solution. A black spot due to Bi metal.
	Residue: Red - Se. Dissolve the residue by heating with 1 cm ³ of conc. HNO ₃ . Neutralise by adding amm. carbonate solution dropwise till effervescence stops. Divide the solution into three parts. 1) To one part, add BaCl ₂ solution. A white precipitate of BaSeO ₃ is formed. 2) To the second part, add CuSO ₄ solution. A bluish green precipitate of CuSeO ₃ is formed. 3) Spot test: Place a drop of the third portion on a spot plate and add a drop of thiourea solution. A red precipitate of Se is formed. Presence of Se (IV) is confirmed.	Centrifugate: Dilute with equal volume of water. Add 1 cm ³ of KI solution and a drop of sodium sulphite (Na ₂ SO ₃) solution. If a precipitate is formed, centrifuge Residue: Black - Te Dissolve in 2 cm ³ of cold dilute HCl. Divide the solution into three parts. 1) Neutralise one portion by adding amm. carbonate solution dropwise till effervescence stops. Add BaCl ₂ solution. A white precipitate of BaTeO ₃ is formed. 2) To the second part, add excess of KI solution. A red colour is produced due to formation of [TeI ₆] ²⁻ . 3) Spot test: Place a drop of the third portion on a spot plate and add a drop of 50% hypophosphorus acid. Evaporate. A black stain due to Te is seen. Presence of Te (IV) is confirmed.	

Table 3

The Group-III A (Iron Group) residue is washed with 2% amm. nitrite (NH_4NO_2) solution and dissolved in the minimum quantity of dilute HCl. Prepare a mixture containing 2 cm ³ of 30% H_2O_2 solution and a fresh pellet of NaOH dissolved in 2 cm ³ of water. Add the weakly acidic test solution into it and boil for 5 minutes. Centrifuge.							
Residue: White – $\text{TiO}_2 \cdot x\text{H}_2\text{O}$ or $\text{ZrO}_2 \cdot x\text{H}_2\text{O}$ or $\text{ThO}_2 \cdot x\text{H}_2\text{O}$ or $\text{MnO}_2 \cdot x\text{H}_2\text{O}$; Brown – $\text{Fe}(\text{OH})_3$; Yellow – $\text{CeO}_3 \cdot x\text{H}_2\text{O}$. Wash the residue with hot 2% amm. nitrite (NH_4NO_2) solution (see instruction 9). Dissolve by boiling with 5 cm ³ of dilute HCl and do the following tests as appropriate:				Centrifugate: May contain CrO_4^{2-} , $[\text{Al}(\text{OH})_4]^-$, VO_3^- or $\text{U}_2\text{O}_7^{2-}$. Transfer to a small beaker and acidify with 2 cm ³ of conc. HNO_3 and dilute with 5 cm ³ of water. Boil and concentrate to half volume. Add 2 cm ³ of lead nitrate solution and a pinch of solid amm. acetate. Boil and centrifuge.			
<p>1) To 0.5 cm³ of the solution, add a drop of pot. (or amm.) thiocyanate (KCNS) solution. Red colour of FeCNS indicates Fe(III).</p> <p>Spot test: Place a drop of the solution on a drop reaction paper. Add a drop of pot. Ferrocyanide solution. A blue spot of ferric ferrocyanide indicates Fe.</p> <p>Presence of Fe is confirmed.</p>	<p>2) To 1 cm³ of the solution, add a few drops of H_2O_2 solution. Orange colour indicates Ti. White precipitate indicates Zr.</p> <p>Spot test: Place a drop of solution on a drop reaction paper. Add a drop of catechol solution. A yellow or orange spot indicates Ti.</p> <p>Presence of Ti (IV) is confirmed.</p> <p>Spot test: Place a drop of solution on a spot plate. Add a drop of Alizarin-S solution and a drop of conc.HCl. A red precipitate indicates Zr.</p> <p>Presence of Zr(IV) is confirmed.</p>	<p>3) To 1 cm³ of the solution, add a few drops of saturated oxalic acid solution. White precipitate indicates Th or Ce. Centrifuge and discard supernatant. Boil residue with 5 cm³ of saturated amm. oxalate solution. If there is a residue, centrifuge.</p> <p>Residue: Ce(III)oxalate.</p> <p>Boil residue with 2 cm³ of NaOH solution. A yellow precipitate of $\text{Ce}(\text{OH})_4$ is obtained.</p> <p>Spot test: Retrieve a little of the above precipitate and dissolve it in two drops of conc.HNO_3. Place a drop of solution on a spot plate and add a drop of pot. Ferrocyanide solution. A white precipitate of thorium ferrocyanide is formed.</p> <p>Presence of Th (IV) is confirmed.</p> <p>Presence of Ce(IV) is confirmed.</p>	<p>4) To 1 cm³ of the solution, add a few drops of conc. H_2SO_4 and evaporate till fumes. Cool, dilute and add two drops of conc. HNO_3 and a pinch of PbO_2. Boil and allow to stand. Purple supernatant (MnO_4^-) indicates Mn.</p> <p>If Mn is indicated, add excess of NH_3 solution into the remaining solution and pass H_2S gas. A pink precipitate of MnS. Confirm as in Table 4.</p> <p>Spot test: Place a drop of the aqueous layer on a spot plate and add a drop of CuSO_4 solution. A green (metavanadate) or yellow (pyrovanadate) precipitate.</p> <p>Presence of V(IV) is confirmed.</p>	<p>Residue: Yellow - PbCrO_4 or $\text{Pb}(\text{VO}_3)_2$.</p> <p>Dissolve in 5 cm³ of dilute HNO_3 by heating and transfer into a test tube. Cool well and add 5 cm³ of amyl alcohol and two drops of conc. H_2O_2 solution. Close the mouth of the tube and mix well by inverting a few times. Allow the two layers to separate. A blue colour in the upper layer due to CrO_5 indicates Cr. A brownish red colour in the lower layer due to VO_2^{3+} indicates V.</p> <p>Spot test: Place a drop of the amyl alcohol layer on a spot reaction paper and evaporate off the alcohol by warming. Place a drop of diphenyl carbazide reagent on it. A blueviolet colour is seen.</p> <p>Presence of Cr(III) is confirmed.</p>	<p>Centrifugate: May contain Al^{3+}, or UO_2^{2+} and excess of Pb^{2+}. Pass H_2S through the solution to precipitate all the PbS. Centrifuge. Wash the precipitate with 2 cm³ of water and combine the centrifugates. Boil to expel all the H_2S and concentrate. Neutralise with NH_3 solution and add 5 cm³ of amm. carbonate solution. Boil for 5 minutes. Cool and centrifuge.</p> <p>Residue: White – $\text{Al}(\text{OH})_3$.</p> <p>Dissolve the residue in 1 cm³ of dilute HCl. Add NaOH solution dropwise with shaking till excess. A white precipitate dissolving in excess indicates Al. Do the spot test using this solution.</p> <p>Spot test: Place a drop of the solution on a spot plate and add a drop of Alizarin-S reagent. Add drops of acetic acid till violet colour is appears. A red precipitate.</p> <p>Presence of Al(III) is confirmed.</p>	<p>Centrifugate: May contain $[\text{UO}_2(\text{CO}_3)_3]^{4-}$.</p> <p>Evaporate to a small volume, acidify with acetic acid and divide into two parts.</p> <p>1) To one portion, add NaOH solution. Yellow precipitate of $\text{Na}_2\text{U}_2\text{O}_7$ indicates U.</p> <p>Spot test: Place a drop of the solution on a spot plate and add pot. ferrocyanide solution and then NaOH solution. A brown precipitate of $(\text{UO}_2)_2[\text{Fe}(\text{CN})_6]$ changing to yellow in NaOH indicates U.</p> <p>Presence of U(VI) is confirmed.</p>	

Table 4

<p>The Group III B (Zinc Group) residue may contain CoS (black), NiS (black), MnS (pink) or ZnS (grey). Wash the precipitate with 2 cm³ of water containing a little NH₃, NH₄Cl and H₂S (see instruction 9). Centrifuge and reject washings. Transfer the precipitate into a small beaker and add 5 cm³ of dilute HCl followed by 5 cm³ of water. Stir well and allow to stand for 5 minutes. If there is a residue, centrifuge.</p>			
<p>Residue: Black – CoS or NiS. Dissolve in 4 cm³ of aqua regia and transfer to a china dish. Evaporate carefully by heating over a wire gauze to dryness. Do the following tests as appropriate:</p>		<p>Centrifugate: May contain Mn²⁺ or Zn²⁺. Boil down to half volume to remove all H₂S. Add 2 cm³ of NaOH solution and two drops of conc. H₂O₂ solution. Boil for 2 minutes and if there is a residue, centrifuge.</p>	
<p>Blue residue: Indicates Co²⁺.</p> <p>Dissolve residue in 2 cm³ of water. Pink solution indicates Co²⁺.</p> <p>Carry out the following tests using this solution.</p> <p>1) To 1 cm³ of the solution, add NaOH solution dropwise with shaking till excess and warm. A blue precipitate of Co(OH)NO₃ appears first, which changes to pink Co(OH)₂.</p> <p>2) Spot test: Place a drop of the solution on a spot plate and two drops of amm. (or pot.) thiocyanate solution. A blue colour appears due to the formation of [Co(SCN)₄]²⁻.</p> <p>Presence of Co²⁺ is confirmed.</p>	<p>Yellow residue: Indicates Ni²⁺.</p> <p>Dissolve residue in 2 cm³ of water. Green solution indicates Ni²⁺.</p> <p>Carry out the following tests using this solution.</p> <p>1) To 1 cm³ of the solution, add NaOH solution dropwise with shaking till excess and warm. A green precipitate of Ni(OH)₂ appears which is insoluble in excess.</p> <p>2) Spot test: Place a drop of the solution on a spot plate. Add one drop each of dimethylglyoxime reagent and NH₃ solution. A red colour or precipitate of nickel dimethyl glyoxime complex is formed.</p> <p>Presence of Ni(II) is confirmed.</p>	<p>Residue: Dark brown MnO₂.xH₂O.</p> <p>Dissolve by boiling with 2 cm³ of dilute sulphuric acid. Pink solution indicates Mn²⁺.</p> <p>Carry out the following tests using this solution.</p> <p>1) To 1 cm³ of the solution, add NaOH solution dropwise with shaking till excess and warm. A white precipitate of Mn(OH)₂ appears which is insoluble in excess. The precipitate rapidly becomes brown when exposed to air due to formation of MnO(OH)₂.</p> <p>2) Spot test: Place a drop of the solution on a spot plate. Add one drop of conc. HNO₃ and a small crystal of solid sodium bismuthate. A purple colour is produced due to formation of permanganate ions.</p> <p>Presence of Mn(II) is confirmed.</p>	<p>Centrifugate: May contain Na₂ZnO₂.</p> <p>1) To 1 cm³ of the solution, add dilute H₂SO₄ solution drop wise with shaking till excess. A white precipitate of Zn(OH)₂ forms, which dissolves in excess as ZnSO₄. (Use this solution for the following spot test).</p> <p>2) Spot test: Place a drop of the above solution on a spot plate. Add one drop each of CuSO₄ solution and amm. Mercuriothiocyanate reagent. A violet precipitate consisting of the mixed mercuriothiocyanates of Cu and Zn is formed.</p> <p>Presence of Zn(II) is confirmed</p>

Table 5

<p>Transfer the Group IV (Barium Group) residue into a small beaker and dissolve in 5 cm³ of dilute acetic acid by heating on a water bath. When completely dissolved, add pot. chromate (K₂CrO₄) solution dropwise while stirring the hot solution, till a faint yellow colour persists in the supernatant indicating excess of the reagent. If a precipitate forms, boil, allow to cool and centrifuge.</p>		
<p>Residue: Yellow - BaCrO₄. Dissolve the residue by heating with 1 cm³ of conc.HCl and transfer into a china dish. Evaporate to dryness.</p> <p>Flame test: Take a speck of the dry residue at the tip of a charred match stick and introduce into a colourless Bunsen flame near the base. The flame is coloured an apple green, indicating Ba.</p> <p>Extract the remaining residue with 1 cm³ of dilute HCl and divide into two parts. To one part, add dilute H₂SO₄ solution. A white heavy precipitate of BaSO₄ is obtained.</p> <p>Spot test: Place a drop of the second part on a drop reaction paper and add a drop of sodium rhodizonate reagent. A red stain is produced due to the formation of barium rhodizonate.</p> <p>Presence of Ba(II) is confirmed.</p>	<p>Centrifugate: Transfer into a small beaker. Add 2 cm³ of saturated amm. sulphate solution and 0.5 cm³ of sodium thiosulphate solution. Boil and reduce to half its volume. If a precipitate forms, cool and centrifuge</p>	
	<p>Residue: White – SrSO₄. Dissolve the residue by heating with 1 cm³ of conc. HCl and transfer into a china dish. Evaporate to dryness.</p> <p>Flame test: Take a speck of the dry residue at the tip of a charred match stick and introduce into a colourless Bunsen flame near the base. The flame is coloured crimson, indicating Sr.</p> <p>Extract the remaining residue with 1 cm³ of dilute HCl and divide into two parts. To one part, add saturated CaSO₄ solution. A scanty white precipitate of SrSO₄ is obtained.</p> <p>Spot test: Place a drop of the second part on a drop reaction paper and add a drop of sodium rhodizonate reagent. A reddish brown stain is produced due to the formation of strontium rhodizonate.</p> <p>Presence of Sr(II) is confirmed.</p>	<p>Centrifugate: May contain Ca as complex.</p> <p>Transfer to a small beaker and make alkaline by adding NH₃ solution dropwise with shaking till smell persists. Add 2 cm³ of amm. Oxalate solution. A white precipitate of CaC₂O₄ appears slowly.</p> <p>Centrifuge and discard centrifugate. Dissolve the residue by heating with 1 cm³ of conc. HCl and transfer into a china dish. Evaporate to dryness.</p> <p>Flame test: Take a speck of the dry residue at the tip of a charred match stick and introduce into a colourless Bunsen flame near the base. The flame is coloured brick red, indicating Ca.</p> <p>Spot test: Extract the remaining residue with 1 cm³ of dilute acetic acid. Place a drop of it on a spot plate and add two drops of picrolinic acid reagent. A white precipitate of calcium picrolinate is produced.</p> <p>Presence of Ca(II) is confirmed.</p>

Table 6	Table 7 (Other Radicals)
Dissolve the Ppt in 2 cm ³ of dilute HCl . The Solution is used as follows	The Cations of this group are generally water soluble, hence they are detected from the water extract. Water is prepared by boiling 0.2g of the mixture with 1 cm ³ of water. It is centrifuge after cooling and the centrifugate is used as follows.
<p style="text-align: center;">For magnesium</p> <ol style="list-style-type: none"> Two drops of the solution in a test tube + 4N NH₄OH, and then NH₃ solution drop wise till smell persists. Add 1 cm³ 3% Oxine reagent in CHCl₃. Shake well and centrifuge – Yellowish Green layer Two drops of solution in a test tube + 0.5cm³ of 10% NaOH + 2-3 drops of 0.1% Titan Yellow Reagent, Centrifuge it – Red Ppt or Colour. Two drops of solution in a test tube + 2 drops of 1N NaOH Solution + 2 drops of 0.001 % p-nitrobenzene azo resorcinol (Magneson-I) reagent – A blue colour or precipitate is obtained (which is different from the violet colour of the reagent). This is due to adsorption of the dye on the precipitated Mg(OH)₂. 2 drops of solution + 3-4 drops of freshly prepared hypo iodide reagent – reddish brown Ppt. <p style="text-align: center;">Presence of Mg(II) is confirmed.</p>	<p style="text-align: center;">For lithium</p> <ol style="list-style-type: none"> Flame test: To two drops of the test solution in a china dish, add a drop of conc. HCl and evaporate to dryness. Take a speck of the dry residue at the tip of a charred match stick and introduce into a colourless Bunsen flame near the base. The flame is coloured carmine red, indicating Li. Spot test: Place a drop of the test solution on a spot plate. Add a drop of NaCl solution and a drop of ferric periodate reagent. A white precipitate of KLiFe[IO₆] is formed. <p style="text-align: center;">Presence of Li(I) is confirmed.</p> <p style="text-align: center;">For Potassium</p> <ol style="list-style-type: none"> Take 5-6 drops of water extract and add 0.5 cm³ of 4N NaOH solution to it. Boil strongly to remove NH₄⁺ as NH₃ gas, cool the solution and used as follows Acidify 2-3 drops of solution with 4N CH₃COOH and add 3-4 drops of 5% Na₂Co(NO₂)₆ solution to it. shake well and allow it to stand for about 10-15 sec – A yellow Crystalline Ppt , K⁺ Present <p>C.T.</p> <ol style="list-style-type: none"> 3 drops of the solution (free from NH₃) are treated with 4-5 drops of saturated picric acid solution. On shaking Well a - A yellow Crystalline Ppt 2 drops of the solution (free from NH₄⁺) + 3-4 drops of 50% EtOH + 4-5 drops of saturated tartaric acid solution – White Ppt <p>Spot Test : A drop of solution on a paper containing 1% Dipicryl amine reagent – orange red spot developed</p> <p style="text-align: center;">For Sodium</p> <p>Flame Test : Perform flame test with the mixture- Golden Yellow Flame</p> <p style="text-align: center;">Na⁺ Confirm</p>

Report:

The cations in the given solution are: (1) _____ (2) _____ (3) _____ and (4) _____

Appendix – 1

Reagents required for Inorganic Qualitative Analysis

General: (2 molar) HCl, NH₃, (NH₄)₂CO₃, NaOH, K₂CrO₄, Na₂S₂O₃, KI, FeSO₄, SnCl₂, HOAc, Amm. polysulphide, Na₂CO₃, Amm. acetate, MgNO₃, AgNO₃, amm. molybdate, CuSO₄, BaCl₂, pot. ferrocyanide, mercuric chloride, Pb(NO₃)₂, amm. fluoride,

Conc.: HCl, H₂SO₄, HNO₃, NH₃, H₂O₂, HOAc,

Saturated: NaHTartrate (or tartaric acid), Na hexanitritocobaltate, CaSO₄solution, Na₂HPO₄solution, oxine reagent (Mg),

Solid: NH₄Cl, KNO₂, KClO₃, sod.sulphite, iron filings, oxalic acid, amm. nitrite, PbO₂, amm. acetate, Amm. (or Pot.) thiocyanate, sodium bismuthate

Spot test

Nessler's reagent (for ammonium)

Pot. Nitrite (Hg)

K₂CrO₄ (Ag)

SnCl₂ (W, Mo)

Amm.sulphide (Pb)

KI (Tl)

Pot. Thiocyanate (Hg, Mo, Co)

Thiourea (Bi, Se)

Tartaric acid (Cu)

Cadion-2B (Cd)

AgNO₃ (As)

Hypophosphorus acid (Te)

Bismuth nitrate (Sn)

Rhodamine B (Sb)

Catechol (Ti)

Alizarin-S (Zr, Al)

Anthranilic acid (Ce)

Pot. ferrocyanide (Th, U)

CuSO₄ (V)

diphenylcarbazide (Cr)

dimethyl glyoxime (Ni)

sodium bismuthate (Mn)

amm.mercurothiocyanate (Zn)

sodium rhodizonate (Ba, Sr)

picrolinic acid reagent (Ca)

magneson reagent (Mg)

ferric periodate reagent (Li)

Appendix – 2

Procedure for preparation of reagents for M.Sc. inorganic analysis.

[Cation solutions for analysis are generally 5% aqueous solutions of their nitrates. When nitrates are not easily available commercially, some other soluble salt is used; for example ammonium molybdate for molybdenum, lithium carbonate dissolved in dil.HNO₃ for lithium etc. About 2.5cm³ of each of the four cations are mixed together and given. Care should be taken to avoid precipitation of other ions in the mixture if other anions like chloride or sulphate or phosphate are used.]

General (1 cm³ = 1 mL, 1 dm³ = 1 L) Keep all reagent bottles stoppered!

1. **Acetic acid (dilute)** - Dilute 114 cm³ of glacial acetic acid to 1 dm³ with distilled water.
2. Ammonium carbonate solution – Dissolve 96 g ammonium carbonate in distilled water and dilute to 1 dm³.
3. **Ammonium fluoride solution** - dissolve 1 g NH₄F in 250 cm³ of distilled water.
4. Ammonium molybdate solution – Dissolve 11 g crystalline ammonium molybdate in a mixture of 15 cm³ conc. NH₃ and 10 cm³ water. Add 30 g ammonium nitrate and after complete dissolution, dilute to 250 cm³ with distilled water.
5. **Ammonium polysulphide (yellow ammonium sulphide) solution** – Dilute 140 cm³ of conc. NH₃ solution with distilled water to 1 dm³ and divide into two equal portions. Saturate one portion with H₂S gas (use fume cupboard!). Then add the second portion and mix. Add 32 g sulphur and heat gently until dissolved to give a yellow solution.
6. **Ammonia solution (dilute)** - dilute 140 cm³ of concentrated NH₃ solution to 1 dm³ with distilled water.
7. Barium chloride solution - dissolve 15 g BaCl₂.2H₂O in 250 cm³ of distilled water.
8. **Copper sulphate solution** - dissolve 15 g CuSO₄.5H₂O in 250 cm³ of distilled water.
9. Ferrous sulphate solution - Add 5 drops of conc. H₂SO₄ in 250 cm³ of distilled water and dissolve 35 g FeSO₄.7H₂O in it.
10. **Hydrochloric acid (dilute)** – dilute 175 cm³ of concentrated acid to 1 dm³ with distilled water.
11. Lead nitrate solution - dissolve 20 g PbNO₃ in 250 cm³ of distilled water.
12. **Mercuric chloride solution** - dissolve 4 g HgCl₂ in 250 cm³ of distilled water.
13. Potassium ferrocyanide solution - dissolve 2.5 g K₄Fe(CN)₆.3H₂O in 250 cm³ of distilled water.
14. **Potassium chromate solution** - dissolve 20 g K₂CrO₄ in 1 dm³ of distilled water.
15. Potassium iodide solution - dissolve 8 g KI in 250 cm³ of distilled water.
16. **Silver nitrate solution** - dissolve 5 g AgNO₃ in 250 cm³ of distilled water. (Dark bottle).
17. Sodium carbonate solution – dissolve 13 g anhydrous Na₂CO₃ in 250 cm³ of distilled water.
18. **Sodium hydroxide solution** – dissolve 20 g NaOH pellets in 250 cm³ of distilled water.
19. Sodium thiosulphate solution - dissolve 6 g Na₂S₂O₃.5H₂O in 250 cm³ of distilled water.
20. **Stannous chloride solution** – Add 25 cm³ of conc. HCl to 25 cm³ of distilled water, mix and cool. Dissolve 14 g SnCl₂.2H₂O and dilute to 250 cm³. Place a granule of tin metal at the bottom of the bottle to prevent oxidation.

Appendix – 3

Procedure for preparation of special reagents for spot tests for inorganic analysis.

1. **Alizarin-S reagent** - dissolve 100 mg of Alizarin-S in 100 cm³ distilled water.
2. **Amm. (or Pot.) thiocyanate solution*** – Dissolve 200 mg of the salt in 1 cm³ of acetone (*prepare only before use).
3. **Amm.mercuriothiocyanate*** - Dissolve a crystal of amm. thiocyanate and a crystal of mercuric chloride in 1 cm³ water (*prepare only before use).
4. **Amm.sulphide solution –**
Dilute 14 cm³ of conc. NH₃ solution with distilled water to 100 cm³ and divide into two equal portions. Saturate one portion with H₂S gas. Then add the second portion and mix. The solution should be colourless.
5. **Anthranilic acid reagent** – Dissolve 5 g anthranilic acid in 100 cm³ ethanol.
6. **Bismuth nitrate solution** – To 50 cm³ of distilled water, add 5 cm³ of conc. HNO₃ cautiously. Dissolve 8 g Bi(NO₃)₃.5H₂O and dilute to 100 cm³.
7. **Cadion 2B reagent** – dissolve 20 mg of Cadion 2B in 100 cm³ ethanol and add a drop of NaOH solution.
8. **Catechol solution** - dissolve 10 g of catechol in 100 cm³ distilled water.
9. **Dimethyl glyoxime reagent** – Dissolve 1 g dimethyl glyoxime in 100 cm³ ethanol.
10. **Diphenylcarbazine*** - Mix a small crystal of diphenylcarbazine with two drops of glacial acetic acid and add 1 cm³ ethanol (*prepare only before use).
11. **Ferric periodate reagent** - Dissolve 11 g KOH in 100 cm³ distilled water. Add 2 g pot. periodate and stir to dissolve well. Then add 250 mg FeCl₃ and dissolve.
12. **Hypophosphorus acid** – Pour 20 cm³ of commercial HPO₂ into 80 cm³ of distilled water and mix.
13. **Magneson reagent** – Dissolve 2 g NaOH in 100 cm³ distilled water. Dissolve 500 mg magneson in it.
14. **Nessler's reagent** –
Dissolve 5 g of KI in 5 cm³ of ammonia-free distilled water (solution *a*). Dissolve 3 g mercuric chloride in 50 cm³ of ammonia-free distilled water (solution *b*). Add *b* to *a* dropwise with shaking till a slight, permanent precipitate appears. To this, add another solution containing 20 g KOH in 50 cm³ of ammonia-free distilled water. Dilute to 100 cm³ and keep overnight in a stoppered bottle. Decant the clear liquid into a reagent bottle and keep tightly stoppered, away from ammonia.
15. **Picrolinic acid reagent*** - Shake 200 mg of picrolinic acid with 1 cm³ water and use the clear supernatant (*prepare only before use).
16. **Pot. nitrite solution*** – Dissolve 5 g KNO₂ in 5 cm³ of distilled water (*prepare only before use).
17. **Rhodamine B reagent** - dissolve 10 mg of Rhodamine B in 100 cm³ distilled water.
18. **Sodium rhodizonate*** - Dissolve a crystal of sodium rhodizonate in 1 cm³ water (*prepare only before use).
19. **Tartaric acid solution** – Dissolve 15 g tartaric acid in 100 cm³ distilled water (fungus growth after a few weeks!).
20. **Thiourea solution** - To 50 cm³ distilled water, add 43 cm³ conc. HCl. Allow to cool and dissolve 5 g thiourea in it. Dilute to 100 cm³ with distilled water.