GUIDE FOR M.Sc. INORGANIC CATION MIXTURE ANALYSIS

For use by students of M.Sc. Chemistry – Semesters 1 & 2, Mahatma Gandhi University.
(2001 admission onwards)

Prepared by:

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M.Sc. Chemistry Practical Syllabus for semesters 1 & 2

CH-205 INORGANIC CHEMISTRY – PRACTICALS-I (60 + 60 hours)

A – Qualitative analysis

I. Separation and identification of four metal ions including two less familiar elements such as Tl, W, Se, Mo, Ce, Th, Ti, Zr, V, U and Li. (Na, K and eliminating anions not to be given. A minimum of 5 mixtures containing 5 different rare ions have to be analysed by a student)

B – Quantitative analysis

II. Complexometric titration for the estimation of hardness of water, Zn, Mg, Ca, Ni ions.

III. Colourimetric estimation of Fe, Cu, Ni, Mn, Cr, NH_4^+, phosphate and nitrate ions.

References

1. Vogel – A Text Book of Qualitative Inorganic Analysis – Longman
2. Kolthoff & Stenger – Volumetric Analysis – Intersience
3. Vogel – A Text Book of Quantitative Inorganic Analysis – Longman
4. Kolthoff & Sandell - Text Book of Qualitative Inorganic Analysis.
5. G. Schwarzen Back "Complexometric Titration" Interscience.

Note to Examiners:

1. Candidates may be asked to report four metal ions present in the given mixture.
2. While reporting the scheme of analysis the student is expected to indicate the chemistry involved in the relevant reactions.
3. The candidates may be asked to give the procedure for the quantitative analysis giving the chemistry behind the experiments.
4. Each student has to carry out I, II and III experiments for the practical examination.

SCHEME FOR SEPARATION AND IDENTIFICATION OF INORGANIC CATIONS IN SOLUTION

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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 (NH_4^+), Silver (Ag^+), Mercurous (Hg^+), Mercuric (Hg^{2+}), Lead (Pb^{2+}), Bismuth (Bi^{3+}), Cupric (Cu^{2+}), Cadmium (Cd^{2+}), Arsenic (As^{3+}), Antimony (Sb^{3+}), Stannous (Sn^{2+}), Stannic (Sn^{4+}), Aluminum (Al^{3+}), Chromic (Cr^{3+}), Ferrous (Fe^{2+}), Ferric (Fe^{3+}), Manganous (Mn^{2+}), Cobalt (Co^{2+}), Nickel (Ni^{2+}), Zinc (Zn^{2+}), Calcium (Ca^{2+}), Strontium (Sr^{2+}), Barium (Ba^{2+}), Magnesium (Mg^{2+}).

(b) Thallium (Tl^+), Tungsten as tungstate (WO_4^{2-}), Selenium as selenite (SeO_3^{2-}) or selenate (SeO_4^{2-}), Tellurium as tellurite (TeO_3^{2-}) or tellurate (TeO_4^{2-}), Molybdenum as molybdate (MoO_4^{2-}), Cerium (Ce^{4+}), Thorium (Th^{4+}), Titanium (Ti^{4+}), Zirconium as zirconyl (ZrO^{2+}), Vanadium as metavanadate (VO_3^{2-}), Uranium as uranyl (UO_2^{2+}), Lithium (Li^+).

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

CATION GROUPS AND REAGENTS

Group	Reagent	Cations (some metals as more stable oxyanions)
Group I	dil. HCl (2M)	Hg^+, Ag^+, Pb^{2+}, Tl^+, WO_4^{2-}.
Group II A B	dil. HCl + H_2S 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	NH_4Cl + NH_4OH 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 IV	NH_4Cl + NH_4OH+ H_2S gas	Co^{2+}, Ni^{2+}, Mn^{2+}, Zn^{2+}.
Group V	NH_4Cl + NH_4OH + $(NH_4)_2CO_3$ solutions (2M)	Ca^{2+}, Sr^{2+}, Ba^{2+}.
Group VI	No common reagent	NH_4^+, Mg^{2+}, Li^+.

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 cm^3 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 cm^3 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, $(NH_4)^+$

***Note**: Do the tests for ammonium using the original solution given.*

Tests	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. $NH_4^+ + OH^- \rightarrow NH_3\uparrow + H_2O$
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_4Cl.
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. $H_2C_4H_4O_6 + NH_4^+ \rightarrow NH_4HC_4H_4O_6\downarrow + H^+$
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). $3\ NH_4^+ + [Co(NO_2)_6]^{3-} \rightarrow (NH_4)_3[Co(NO_2)_6]\downarrow$
<u>Spot test</u>: Mix a drop of the test solution with a drop of sodium hydroxide solution on a watch glass. Place a microdrop 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. (*Note: Various formulae have been reported for the product by different authors*).

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.

<table>
<tr><td colspan="6">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.</td></tr>
<tr><td rowspan="5">**Residue**:
Chlorides of Group I metals or $H_2WO_4.H_2O$.
White - Hg^+, Ag^+, Pb^{2+}, Tl^+, or WO_4^{2-}.
Examine as given in Table 1.</td><td colspan="5">**Centrifugate**: Add a drop of H_2O_2 and dilute the solution to double its volume with water. Heat nearly to boiling and saturate with H_2S gas. Shake well. If a precipitate forms, continue passing H_2S till no more precipitate is formed and smell of H_2S persists. Centrifuge.</td></tr>
<tr><td rowspan="4">**Residue**:
Sulphides of Group II metals.
Black – Hg^{2+}, Pb^{2+}, Bi^{3+} or Cu^{2+}.
Yellow – Cd^{2+}, Sn^{4+}, Se or As^{3+}.
Orange red - Sb^{3+}.
Brown -Te, Sn^{2+} or Mo^{6+}.
Examine as given in Table 2.</td><td colspan="4">**Centrifugate**: Boil down to half its volume in a small beaker till no smell of H_2S remains. Add 1 cm^3 of concentrated HNO_3 (to oxidize any Fe^{2+} to Fe^{3+}). Evaporate till almost dry and dilute to 5 cm^3 with water. Add 2 g of solid NH_4Cl and heat to boiling. Add dilute NH_3 solution drop by drop with shaking till a strong smell of ammonia persists. Heat to boiling and centrifuge.</td></tr>
<tr><td rowspan="3">**Residue**:
Hydroxides or oxides of group III metals.
White - Al^{3+}, VO_3^{2-}, Ti^{4+}, ZrO^{2+} or Th^{4+};
White, slowly turning brown - Ce^{4+}
Bluish green - Cr^{3+};
Brown - Fe^{3+};
Yellow - UO_2^{2+}.
Examine as given in Table 3.</td><td colspan="3">**Centrifugate**: Evaporate to reduce volume. Add dilute NH_3 solution drop by drop with shaking till a strong smell of ammonia persists. Saturate with H_2S gas. Shake well. If a precipitate forms, continue passing H_2S till no more precipitate is formed and smell of H_2S persists. Boil. Centrifuge.</td></tr>
<tr><td rowspan="2">**Residue**:
Sulphides of group IV metals.
Black - Co^{2+} or Ni^{2+};
Pink - Mn^{2+};
White - Zn^{2+}.
Examine as given in Table 4.</td><td colspan="2">**Centrifugate**: Evaporate till almost dry and dilute to 5 cm^3 with water. Add dilute NH_3 solution drop by drop with shaking till a strong smell of ammonia persists. Add $(NH_4)_2CO_3$ solution dropwise with stirring till precipitation is complete.</td></tr>
<tr><td>**Residue**:
Carbonates of group V metals.
White - Ca^{2+}, Sr^{2+} or Ba^{2+}.
Examine as given in Table 5.</td><td>**Centrifugate**: May contain Mg^{2+} or Li^+ (group VI metals)
Evaporate in a china dish till dry and the fumes stop. Cool and extract residue with 5 cm^3 of water.
Examine as given in Table 6.</td></tr>
</table>

Table 1.

<table>
<tr><td colspan="5">The group I residue is washed using 2 cm^3 of dilute HCl (see instruction 9) and then with 2 cm^3 of water. Discard the washings. Boil residue in the centrifuge tube with 2 cm^3 of water and centrifuge while hot. Decant the centrifugate into a test tube. Repeat extraction with 2 cm^3 boiling water and combine the centrifugates.</td></tr>
<tr><td colspan="3">Residue: White – Hg_2Cl_2 or AgCl. Yellow - H_2WO_4.
Boil the residue with 5 cm^3 of dilute NH_3 solution and centrifuge.</td><td colspan="2">Centrifugate: May contain Pb^{2+}or Tl^+, which may crystallize on cooling. Carefully add 1 cm^3 of conc. H_2SO_4 and evaporate till fumes are seen. Cool and dilute carefully to 5 cm^3 with water. If a precipitate is formed, centrifuge.</td></tr>
<tr><td rowspan="2">Residue: Black precipitate, consisting of a mixture of $Hg(NH_2)Cl$ and Hg.

Dissolve the residue by heating with 2 cm^3 of aqua regia, cool and do the following tests:

1) To 1 cm^3 of the solution, add NaOH solution in drops with shaking till excess. A black precipitate of Hg_2O is formed.
2) To 1 cm^3 of the solution, add K_2CrO_4 solution and heat. A brown precipitate changing to red Hg_2CrO_4 on heating.
3) <u>Spot test</u>: Place a drop of faintly acid test solution (original) on a drop reaction paper and add a drop of saturated pot. nitrite (KNO_2) solution. A black spot of Hg is produced.

Presence of Hg(I) is confirmed.</td><td colspan="2">Centrifugate: May contain $[Ag(NH_3)_2]$+ or H_2WO_4. Add dilute HCl in drops with shaking till a faint precipitate forms, which is redissolved by adding one drop of dilute NH_3 solution. Add KI solution dropwise with shaking till precipitation, if any, is complete. Centrifuge.</td><td rowspan="2">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_2CrO_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 reprecipitates as golden spangles on cooling.

3) <u>Spot test</u>: 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.

Presence of Pb(II) is confirmed.</td><td rowspan="2">Centrifugate: May contain Tl^+. Add dilute NH_3 solution and boil off excess NH_3. Cool and do the following tests:

1) To 1 cm^3 of the solution, add acetic acid and K_2CrO_4 solution. A yellow precipitate of Tl_2CrO_4 is formed.

2) Flame test: Evaporate 2 cm^3 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.

3) <u>Spot test</u>: Place a drop of the solution on a drop reaction paper and add a drop sod. thiosulphate solution. Add a drop of KI solution over it. A yellow spot of TlI is produced.

Presence of Tl(I) is confirmed.</td></tr>
<tr><td>Residue: Pale yellow precipitate of AgI.

1)To a portion of the above precipitate, add excess of sod. thiosulphate solution. The precipitate dissolves. Presence of Ag^+ confirmed.

2) <u>Spot test</u>: Place a drop of the test solution (original) on a watch glass and add a drop of amm. 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 K_2CrO_4 solution. A red ring is obtained due to formation of Ag_2CrO_4.

Presence of Ag(I) is confirmed.</td><td>Centrifugate: Tungstic acid (H_2WO_4) solution.

Reduce to half its volume by evaporation, cool and do the following tests:

1) To 0.5 cm^3 of the solution, add ferrous sulphate solution. A brown precipitate of ferrous tungstate is formed. Add 1 cm^3 of dilute HCl and boil. The precipitate turns white and then yellow due to formation of tungstic acid.

2) <u>Spot test</u>: Place a drop of the test solution (original) on a drop reaction paper and add a drop of conc. HCl. A drop of saturated $SnCl_2$ solution is placed at the centre of the spot. A blue colour is produced due to formation of tungsten blue, W_2O_5.

Presence of W(VI) is confirmed.</td></tr>
</table>

Table 2.

<table>
<tr><td colspan="2">Wash the group II residue with 2 cm^3 of a solution of ammonium chloride saturated with H_2S and centrifuge. Discard centrifugate. Transfer residue into a small beaker and add 5 cm^3 of yellow ammonium sulphide (amm. polysulphide) solution. Heat to about 50 to 60°C and maintain at this temperature for 5 minutes with stirring. If there is a precipitate, cool and centrifuge.</td></tr>
<tr><td rowspan="2">Residue: Black – HgS, PbS, Bi_2S_3 or CuS. Yellow – CdS.
Analyse as given in Table 2A.</td><td>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 sulpur. If a flocculant precipitate is formed, centrifuge. If not, discard the solution.</td></tr>
<tr><td>Residue: Yellow – SnS_2, Se or As_2S_3. Orange red – Sb_2S_3. Brown -Te, SnS or MoS_3.
Analyse as given in Table 2B.</td></tr>
</table>

Table 2A

<table>
<tr><td colspan="5">The group IIA 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.</td></tr>
<tr><td rowspan="4">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_2CO_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) <u>Spot test</u>: 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.
Presence of Hg(II) is confirmed.</td><td colspan="4">Centrifugate: May contain nitrate of Pb, Bi, Cu or Cd. Add dilute H_2SO_4 till precipitation (if any) is complete. Centrifuge.</td></tr>
<tr><td rowspan="3">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_2CrO_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 reprecipitates as golden spangles on cooling.
3) <u>Spot test</u>: 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.
Presence of Pb(II) is confirmed.</td><td colspan="3">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.</td></tr>
<tr><td rowspan="2">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_2O_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) <u>Spot test</u>: Place a drop of the second part on a spot plate and add a drop of thiourea solution. An intense yellow colour is produced.
Presence of Bi(III) is confirmed.</td><td colspan="2">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.</td></tr>
<tr><td>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. $2\ Cu(SCN)_2 \rightarrow 2\ CuSCN + (SCN)_2$.
3) <u>Spot test</u>: 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.
Presence of Cu(II) is confirmed.</td><td>Divide the second part into three equal portions and do the following tests:
1) To one part, add NaOH solution. A white precipitate of $Cd(OH)_2$ is formed.
2) Through the second portion, pass H_2S. A yellow precipitate of CdS is obtained.
3) <u>Spot test</u>: Place a drop Cadion- 2B reagent on a drop reaction paper. Place a drop from the third part over it, followed by a drop of KOH solution. A bright pink spot surrounded by a blue circle is produced.
Presence of Cd(II) is confirmed.</td></tr>
</table>

Table 2B

<table>
<tr><td colspan="6">Transfer the group IIB 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.</td></tr>
<tr><td colspan="4">**Residue**: Yellow - Se or As_2S_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 $MgNO_3$ solution and stir for 5 minutes. When precipitation is complete, centrifuge.</td><td colspan="2">**Centrifugate**: May contain Sb or Sn as chloride. Boil to expel H_2S. Cool and carry out the following tests using small portions of the solution.</td></tr>
<tr><td rowspan="3">**Residue**: White crystalline $Mg(NH_4)AsO_4.6H_2O$
Shake the residue well with 3 cm^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_3AsO_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_2O_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.</td><td colspan="3">**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_2SO_3) solution. If a precipitate is formed, centrifuge.</td><td rowspan="3">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. Withdraw 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_2Cl_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.
Presence of Sn is confirmed.</td><td rowspan="3">To 1 cm^3 of the solution in a test tube, add just enough NH_3 solution to neutralize. (A slight precipitation may occur). Add a pinch of solid oxalic acid and boil. Pass H_2S through the solution. An orange precipitate of Sb_2S_3 is obtained.
Spot test: Place a drop of the solution on a spot plate and add a minute crystal of sodium nitrite ($NaNO_2$). Then add a drop of rhodamine-B reagent. A blue colour is obtained.
Presence of Sb(II) is confirmed.</td></tr>
<tr><td rowspan="2">**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.</td><td colspan="2">**Centrifugate**: Dilute with equal volume of water. Add 1 cm^3 of KI solution and a drop of sodium sulphite (Na_2SO_3) solution. If a precipitate is formed, centrifuge.</td></tr>
<tr><td>**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.</td><td>**Centrifugate**: Add 1 cm^3 of conc. HCl and boil to remove dissolved SO_2. Divide the solution into three parts.
1) To one part, add pot. ferrocyanide solution. A reddish brown precipitate of Mo ferrocyanide is formed.
2) To the second part, add two drops of ferrous sulphate solution. A blue colour is produced.
3) Spot test: Place a drop of the third portion on a drop reaction paper and add a drop of pot. thiocyanate solution. Then add a drop of $SnCl_2$ solution. A red spot of $[Mo(SCN)_6]^{3-}$ is seen.
Presence of Mo(VI) is confirmed.</td></tr>
</table>

Table 3

<table>
<tr><td colspan="8">The group-III 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.</td></tr>
<tr><td colspan="5">Residue: White – $TiO_2.xH_2O$ or $ZrO_2.xH_2O$ or $ThO_2.xH_2O$ or $MnO_2.xH_2O$; Brown – $Fe(OH)_3$; Yellow – $CeO_3.xH_2O$. 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:</td><td colspan="3">Centrifugate: May contain CrO_4^{2-}, $[Al(OH)_4]^-$, VO_3^- or $U_2O_7^{2-}$. 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.</td></tr>
<tr><td rowspan="2">1) To 0.5 cm^3 of the solution, add a drop of pot. (or amm.) thiocyanate (KCNS) solution. Red colour of FeCNS indicates Fe(III).
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.
Presence of Fe is confirmed.</td><td rowspan="2">2) To 1 cm^3 of the solution, add a few drops of H_2O_2 solution. Orange colour indicates Ti. White precipitate indicates Zr.
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.
Presence of Ti (IV) is confirmed.
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.
Presence of Zr(IV) is confirmed.</td><td colspan="2">3) To 1 cm^3 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 cm^3 of saturated amm. oxalate solution. If there is a residue, centrifuge.</td><td rowspan="2">4) To 1 cm^3 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.
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.
←
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.
Presence of V(IV) is confirmed.</td><td rowspan="2">Residue: Yellow - $PbCrO_4$ or $Pb(VO_3)_2$.
Dissolve in 5 cm^3 of dilute HNO_3 by heating and transfer into a test tube. Cool well and add 5 cm^3 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.
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 blue violet colour is seen.
Presence of Cr(III) is confirmed.
←</td><td colspan="2">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 cm^3 of water and combine the centrifugates. Boil to expel all the H_2S and concentrate. Neutralise with NH_3 solution and add 5 cm^3 of amm. carbonate solution. Boil for 5 minutes. Cool and centrifuge.</td></tr>
<tr><td>Residue: Ce(III)oxalate.
Boil residue with 2 cm^3 of NaOH solution. A yellow precipitate of $Ce(OH)_4$ is obtained.
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 a 5% alcoholic solution of anthranilic acid. A blackish blue precipitate rapidly changing to a brown solution.
Presence of Ce(IV) is confirmed.</td><td>Centrifugate: Acidify 1 cm^3 of the solution with dilute HCl. Thorium oxalate is reprecipitated.
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.
Presence of Th (IV) is confirmed.</td><td>Residue: White – $Al(OH)_3$.
Dissolve the residue in 1 cm^3 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.
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 disappears. A red precipitate.
Presence of Al(III) is confirmed.</td><td>Centrifugate: May contain $[UO_2(CO_3)_3]^{4-}$.
Evaporate to a small volume, acidify with acetic acid and divide into two parts.
1) To one portion, add NaOH solution. Yellow precipitate of $Na_2U_2O_7$ indicates U.
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 $(UO_2)_2[Fe(CN)_6]$ changing to yellow in NaOH indicates U.
Presence of U(VI) is confirmed.</td></tr>
</table>

Table 4

The group IV residue may contain CoS (black), NiS (black), MnS (pink) or ZnS (grey). Wash the precipitate with 2 cm^3 of water containing a little NH_3, NH_4Cl and H_2S (see instruction 9). Centrifuge and reject washings. Transfer the precipitate into a small beaker and add 5 cm^3 of dilute HCl followed by 5 cm^3 of water. Stir well and allow to stand for 5 minutes. If there is a residue, centrifuge.			
Residue: Black – CoS or NiS. Dissolve in 4 cm^3 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:		**Centrifugate**: May contain Mn^{2+} or Zn^{2+}. Boil down to half volume to remove all H_2S. Add 2 cm^3 of NaOH solution and two drops of conc. H_2O_2 solution. Boil for 2 minutes and if there is a residue, centrifuge.	
Blue residue: Indicates Co^{2+}. Dissolve residue in 2 cm^3 of water. Pink solution indicates Co^{2+}. Carry out the following tests using this solution. 1) To 1 cm^3 of the solution, add NaOH solution dropwise with shaking till excess and warm. A blue precipitate of $Co(OH)NO_3$ appears first, which changes to pink $Co(OH)_2$. 2) <u>Spot test</u>: 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 $[Co(SCN)_4]^{2-}$. **Presence of Co^{2+} is confirmed.**	**Yellow residue**: Indicates Ni^{2+}. Dissolve residue in 2 cm^3 of water. Green solution indicates Ni^{2+}. Carry out the following tests using this solution. 1) To 1 cm^3 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. 2) <u>Spot test</u>: Place a drop of the solution on a spot plate. Add one drop each of dimethylglyoxime reagent and NH_3 solution. A red colour or precipitate of nickel dimethylglyoxime complex is formed. **Presence of Ni(II) is confirmed.**	**Residue**: Dark brown – $MnO_2.xH_2O$. Dissolve by boiling with 2 cm^3 of dilute sulphuric acid. Pink solution indicates Mn^{2+}. Carry out the following tests using this solution. 1) To 1 cm^3 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 $MnO(OH)_2$. 2) <u>Spot test</u>: Place a drop of the solution on a spot plate. Add one drop of conc. HNO_3 and a small crystal of solid sodium bismuthate. A purple colour is produced due to formation of permanganate ions. **Presence of Mn(II) is confirmed.**	**Centrifugate**: May contain Na_2ZnO_2. 1) To 1 cm^3 of the solution, add dilute H_2SO_4 solution dropwise with shaking till excess. A white precipitate of $Zn(OH)_2$ forms, which dissolves in excess as $ZnSO_4$. (Use this solution for the following spot test). 2) <u>Spot test</u>: Place a drop of the above solution on a spot plate. Add one drop each of $CuSO_4$ solution and amm. mercurothiocyanate reagent. A violet precipitate consisting of the mixed mercurothiocyanates of Cu and Zn is formed. **Presence of Zn(II) is confirmed.**

Table 5

<table>
<tr><td colspan="3">Transfer the group V residue into a small beaker and dissolve in 5 cm^3 of dilute acetic acid by heating on a water bath. When completely dissolved, add pot. chromate (K_2CrO_4) 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.</td></tr>
<tr><td rowspan="2">**Residue**: Yellow - $BaCrO_4$.
Dissolve the residue by heating with 1 cm^3 of conc. HCl and transfer into a china dish. Evaporate to dryness.
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.
Extract the remaining residue with 1 cm^3 of dilute HCl and divide into two parts. To one part, add dilute H_2SO_4 solution. A white heavy precipitate of $BaSO_4$ is obtained.
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.
Presence of Ba(II) is confirmed.</td><td colspan="2">**Centrifugate**: Transfer into a small beaker. Add 2 cm^3 of saturated amm. sulphate solution and 0.5 cm^3 of sodium thiosulphate solution. Boil and reduce to half its volume. If a precipitate forms, cool and centrifuge.</td></tr>
<tr><td>**Residue**: White – $SrSO_4$.
Dissolve the residue by heating with 1 cm^3 of conc. HCl and transfer into a china dish. Evaporate to dryness.
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.
Extract the remaining residue with 1 cm^3 of dilute HCl and divide into two parts. To one part, add saturated $CaSO_4$ solution. A scanty white precipitate of $SrSO_4$ is obtained.
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.
Presence of Sr(II) is confirmed.</td><td>**Centrifugate**: May contain Ca as complex. Transfer to a small beaker and make alkaline by adding NH_3 solution dropwise with shaking till smell persists. Add 2 cm^3 of amm. oxalate solution. A white precipitate of CaC_2O_4 appears slowly.
Centrifuge and discard centrifugate. Dissolve the residue by heating with 1 cm^3 of conc. HCl and transfer into a china dish. Evaporate to dryness.
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.
Spot test: Extract the remaining residue with 1 cm^3 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.
Presence of Ca(II) is confirmed.</td></tr>
</table>

Table 6

The group VI extract is divided into two equal parts. These are tested as follows, first for magnesium, and then for lithium if magnesium is absent:	
For magnesium	**For lithium**
1) To 1 cm^3 of the solution in a test tube, add a pinch of solid NH_4Cl, and then NH_3 solution dropwise till smell persists. Add 1 cm^3 of saturated Na_2HPO_4 solution and stir with a glass rod. A white crystalline precipitate of $Mg(NH_4)PO_4.6H_2O$ slowly appears. 2) To 1 cm^3 of the solution in a test tube, add a pinch of solid NH_4Cl, and then NH_3 solution dropwise till smell persists. Add 1 cm^3 of oxine reagent (C_9H_6NO) and boil. A yellow precipitate of $Mg(C_9H_6NO)_2.4H_2O$ is formed. 3) <u>Spot test</u>: Place a drop of the test solution on a spot plate. Add a drop of magneson reagent and a drop of NaOH solution to it. 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 $Mg(OH)_2$. **Presence of Mg(II) is confirmed.**	1) To 1 cm^3 of the solution in a test tube, add NH_3 solution dropwise till smell persists. Add 1 cm^3 amm. fluoride solution. A white precipitate of LiF slowly appears. 2) To 1 cm^3 of the solution in a test tube, add NH_3 solution dropwise till smell persists. Add 1 cm^3 amm. carbonate solution. A white precipitate of Li_2CO_3 is formed. 3) <u>Flame test</u>: 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. 3) <u>Spot test</u>: 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 $KLiFe[IO_6]$ is formed. **Presence of Li(I) is confirmed.**

Report:

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

Appendix – 1

Reagents required for Inorgnic Qualitative Analysis

General: (2 molar) HCl, NH_3, $(NH_4)_2CO_3$, NaOH, K_2CrO_4, $Na_2S_2O_3$, KI, $FeSO_4$, $SnCl_2$, HOAc, Amm. polysulphide, Na_2CO_3, Amm. acetate, $MgNO_3$, $AgNO_3$, amm. molybdate, $CuSO_4$, $BaCl_2$, pot. ferrocyanide, mercuric chloride, $Pb(NO_3)_2$, amm. fluoride,

Conc.: HCl, H_2SO_4, HNO_3, NH_3, H_2O_2, HOAc,

Saturated: NaHtartrate (or tartaric acid), Na hexanitritocobaltate, $CaSO_4$ solution, Na_2HPO_4 solution, oxine reagent (Mg),

Solid: NH_4Cl, KNO_2, $KClO_3$, sod.sulphite, iron filings, oxalic acid, amm. nitrite, PbO_2, amm. acetate, Amm. (or Pot.) thiocyanate, sodium bismuthate.

<u>Spot test</u>

Nessler's reagent (for ammonium)
Pot. Nitrite (Hg)
K_2CrO_4 (Ag)
$SnCl_2$ (W, Mo)
Amm. sulphide (Pb)
KI (Tl)
Pot. Thiocyanate (Hg, Mo, Co)
Thiourea (Bi, Se)
Tartaric acid (Cu)
Cadion-2B (Cd)
$AgNO_3$ (As)
Hypophosphorus acid (Te)
Bismuth nitrate (Sn)
Rhodamine B (Sb)
Catechol (Ti)
Alizarin-S (Zr, Al)
Anthranilic acid (Ce)
Pot. ferrocyanide (Th, U)
$CuSO_4$ (V)

diphenyl carbazide (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 amm. molybdate for molybdenum, lithium carbonate dissolved in dil.HNO_3 for lithium etc. About 2.5cm^3 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.]

<u>General</u> (1 cm^3 = 1 mL, 1 dm^3 = 1 L) Keep all reagent bottles stoppered!

Acetic acid (dilute) - Dilute 114 cm^3 of glacial acetic acid to 1 dm^3 with distilled water.
Amm. carbonate solution – Dissolve 96 g amm. carbonate in distilled water and dilute to1 dm^3.
Amm. fluoride solution - dissolve 1 g NH_4F in 250 cm^3 of distilled water.
Amm. molybdate solution – Dissolve 11 g crystalline amm. molybdate in a mixture of 15 cm^3 conc. NH_3 and 10 cm^3 water. Add 30 g amm. nitrate and after complete dissolution, dilute to 250 cm^3 with distilled water.
Amm. polysulphide (yellow amm. sulphide) solution – Dilute 140 cm^3 of conc. NH_3 solution with distilled water to 1 dm^3 and divide into two equal portions. Saturate one portion with H_2S 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.
Ammonia solution (dilute) - dilute 140 cm^3 of concentrated NH_3 solution to 1 dm^3 with distilled water.
Barium chloride solution - dissolve 15 g $BaCl_2.2H_2O$ in 250 cm^3 of distilled water.
Copper sulphate solution - dissolve 15 g $CuSO_4.5H_2O$ in 250 cm^3 of distilled water.
Ferrous sulphate solution - Add 5 drops of conc. H_2SO_4 in 250 cm^3 of distilled water and dissolve 35 g $FeSO_4.7H_2O$ in it.
Hydrochloric acid (dilute) – dilute 175 cm^3 of concentrated acid to 1 dm^3 with distilled water.
Lead nitrate solution - dissolve 20 g $PbNO_3$ in 250 cm^3 of distilled water.
Mercuric chloride solution - dissolve 4 g $HgCl_2$ in 250 cm^3 of distilled water.
Pot. ferrocyanide solution - dissolve 2.5 g $K_4Fe(CN)_6.3H_2O$ in 250 cm^3 of distilled water.
Potassium chromate solution - dissolve 20 g K_2CrO_4 in 1 dm^3 of distilled water.
Potassium iodide solution - dissolve 8 g KI in 250 cm^3 of distilled water.
Silver nitrate solution - dissolve 5 g $AgNO_3$ in 250 cm^3 of distilled water. (Dark bottle).
Sodium carbonate solution – dissolve 13 g anhydrous Na_2CO_3 in 250 cm^3 of distilled water.
Sodium hydroxide solution – dissolve 20 g NaOH pellets in 250 cm^3 of distilled water.
Sodium thiosulphate solution - dissolve 6 g $Na_2S_2O_3.5H_2O$ in 250 cm^3 of distilled water.
Stannous chloride solution – Add 25 cm^3 of conc. HCl to 25 cm^3 of distilled water, mix and cool. Dissolve 14 g $SnCl_2.2H_2O$ and dilute to 250 cm^3. 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.

Alizarin-S reagent - dissolve 100 mg of Alizarin-S in 100 cm^3 distilled water.

Amm. (or Pot.) thiocyanate solution* – Dissolve 200 mg of the salt in 1 cm^3 of acetone (*prepare only before use).

Amm. mercurothiocyanate* - Dissolve a crystal of amm. thiocyanate and a crystal of mercuric chloride in 1 cm^3 water (*prepare only before use).

Amm. sulphide solution – Dilute 14 cm^3 of conc. NH_3 solution with distilled water to 100 cm^3 and divide into two equal portions. Saturate one portion with H_2S gas. Then add the second portion and mix. The solution should be colourless.

Anthranilic acid reagent – Dissolve 5 g anthranilic acid in 100 cm^3 ethanol.

Bismuth nitrate solution – To 50 cm^3 of distilled water, add 5 cm^3 of conc. HNO_3 cautiously. Dissolve 8 g $Bi(NO_3)_3.5H_2O$ and dilute to100 cm^3.

Cadion 2B reagent – dissolve 20 mg of Cadion 2B in 100 cm^3 ethanol and add a drop of NaOH solution.

Catechol solution - dissolve 10 g of catechol in 100 cm^3 distilled water.

Dimethyl glyoxime reagent – Dissolve 1 g dimethyl glyoxime in 100 cm^3 ethanol.

Diphenyl carbazide* - Mix a small crystal of diphenyl carbazide with two drops of glacial acetic acid and add 1 cm^3 ethanol (*prepare only before use).

Ferric periodate reagent - Dissolve 11 g KOH in 100 cm^3 distilled water. Add 2 g pot. periodate and stir to dissolve well. Then add 250 mg $FeCl_3$ and dissolve.

Hypophosphorus acid – Pour 20 cm^3 of commercial HPO_2 into 80 cm^3 of distilled water and mix.

Magneson reagent – Dissolve 2 g NaOH in 100 cm^3 distilled water. Dissolve 500 mg magneson in it.

Nessler's reagent – Dissolve 5 g of KI in 5 cm^3 of ammonia-free distilled water (solution *a*). Dissolve 3 g mercuric chloride in 50 cm^3 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 cm^3 of ammonia-free distilled water. Dilute to 100 cm^3 and keep overnight in a stoppered bottle. Decant the clear liquid into a reagent bottle and keep tightly stoppered, away from ammonia.

Picrolinic acid reagent* - Shake 200 mg of picrolinic acid with1 cm^3 water and use the clear supernatant (*prepare only before use).

Pot. nitrite solution* – Dissolve 5 g KNO_2 in 5 cm^3 of distilled water (*prepare only before use).

Rhodamine B reagent - dissolve 10 mg of Rhodamine B in 100 cm^3 distilled water.

Sodium rhodizonate* - Dissolve a crystal of sodium rhodizonate in 1 cm^3 water (*prepare only before use).

Tartaric acid solution – Dissolve 15 g tartaric acid in 100 cm^3 distilled water (fungus growth after a few weeks!).

Thiourea solution - To 50 cm^3 distilled water, add 43 cm^3 conc. HCl. Allow to cool and dissolve 5 g thiourea in it. Dilute to 100 cm^3 with distilled water.