# I SEMESTER

## PRACTICAL MANUAL FOR FIRST B.Sc. CHEMISTRY

**(w. e. f. 2020 – 2021)**

## PRACTICAL – 1(ANALYSIS OF SALT MIXTURE)


**PREPARED BY**

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# SYLLABUS

## LABORATORY COURSE -I 30hrs (2 H / W)

### Practical-I Analysis of SALT MIXTURE (At the end of Semester-I)

Qualitative inorganic analysis (Minimum of Six mixtures should be analyzed) 50 M

Course outcomes:

At the end of the course, the student will be able to;

- Understand the basic concepts of qualitative analysis of inorganic mixture
- Use glassware, equipment and chemicals and follow experimental procedures in the laboratory
- Apply the concepts of common ion effect, solubility product and concepts related to qualitative analysis

### ANALYSIS OF SALT MIXTURE 50 M

Analysis of mixture salt containing two anions and two cations (From two different groups) from the following:

Anions

Carbonate, Sulphate, Chloride, Bromide, Acetate, Nitrate, Borate, Phosphate.

Cations

Lead, Copper, Iron, Aluminium, Zinc, Nickel, Manganese, Calcium, Strontium, Barium, Potassium and Ammonium.

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# SCHEME OF VALUATION

**Time: 3 Hours** **Maximum Marks: 50 M**

| | |
|---|---|
| Record | Marks:5M |
| Viva-Voce | Marks: 5M |
| Practical | Marks: 40M |

**For Preliminary Examination** **Marks: 8M**

| | |
|---|---|
| **1.** Colour | 1 Mark |
| **2.** Structure | 1 Mark |
| **3.** Solubility | 2 Marks |
| **4.** Action of Heat | 2 Marks |
| **5.** Flame colour test | 2 Marks |

**For anion** **Marks: 16M**

| | |
|---|---|
| **6.** Identification test for anion | 4 Marks |
| **7.** Confirmation tests for anion without soda | 6 Marks |

(For phosphate and sulphate 6 marks shall be added to the marks at item 8 without this test)

| | |
|---|---|
| **8.** Procedure for the preparation of soda extract | 2 Marks |
| **9.** Confirmatory test for anion with soda extract | 4 Marks |

(For carbonate and borate 4 marks shall be added to the marks at item 6 without this test)

**For cation** **Marks:14M**

| | |
|---|---|
| **10.** Identification of the Group | 4 Marks |
| **11.** Colour of the ppt. in the identified Group | 2 Marks |
| **12.** Confirmatory test for cation in the individual Group | 8 Marks |

**Report** 2 Marks

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# QUALITATIVE INORGANIC SALT MIXTURE ANALYSIS

| Experiment | Observation | Inference |
|---|---|---|
| **Colour** | White | Salt mixtures of Cu, Fe, Ni and Mn are absent |
| | Blue or bluish green | May be a copper salt mixture |
| | Brown or yellowish brown | May be a ferric salt mixture |
| | Pale green | May be a salt mixture of ferrous or nickel |
| | Green | May be $CuCO_3$ or $CuCl_2$ |
| | Pale pink | May be a manganese salt mixture |
| **Structure** | Crystalline | May be a hydrated salt mixture |
| | White amorphous (appears as face powder) | May be $ZnCO_3$, $BaCO_3$, $SrCO_3$ $CaCO_3$ |
| | Green amorphous | May be $CuCO_3$ |
| | White deliquescent salt mixture (wet with moisture in air) | May be $ZnCl_2$, $Zn(NO_3)_2$, $Ba(NO_3)_2$ or $CaCl_2$ |
| | Pink deliquescent salt mixture | May be $MnCl_2$ |
| **Action of heat** | Water drops formed on cooler parts of the test tube | Hydrated salt mixture |
| | White coat formed on sides of the test tube (sublimation took place) and ammonia smell evolved. | May be an ammonium halide |
| | | |

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| | | |
|---|---|---|
| | White sublimate formed Without ammonia smell. | May be an aluminium salt mixture |
| | Colourless vapours evolved with vinegar smell. | May be acetate |
| | Light brown vapours evolved with pungent smell. | May be nitrate |
| | Yellow in cold, brown in hot | May be a lead salt mixture |
| | Blue in cold, white in hot. | May be copper sulphate |
| | Light yellow in cold, black in hot. | May be ferric chloride |
| | White in cold, orange yellow in hot. | May be a zinc salt mixture |
| | Green in cold, yellow in hot. | May be a nickel salt mixture |
| **Flame colouration test** On a watch glass a little of the salt mixture was mixed with few drops of conc. HCl and was made as a paste. The paste was exposed to colourless flame by a platinum wire or glass rod. Colour of the flame was observed which indicated presence of the cation | Livid blue (dark blue) | Lead |
| | Bluish green | Copper |
| | Green flashes | Zinc |
| | Apple green(light green) | Barium |
| | Crimson red | Strontium |
| | Brick red | Calcium |
| | Violet | Potassium |

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# IDENTIFICATION TESTS FOR ANIONS

| | | |
|---|---|---|
| 1.**Test with dil. HCl:** To a little of the salt mixture dil. HCl was added | Colourless and odourless gas ($CO_2$) with brisk Effervescence evolved. On passing the gas lime water became milky white ($CaCO_3$). | Carbonate ($CO_3^{2-}$) present |
| | <u>On heating the test tube</u> smell of vinegar ($CH_3COOH$) observed. | Acetate ($CH_3COO^-$) present |
| 2. **Oxalic acid rubbing test for acetate**: On a watch glass a little of the salt mixture, oxalic acid and a few drops of water were taken and rubbed with a glass rod or thumb. | Vinegar smell observed. | Acetate present |
| 3. **Test with conc. $H_2SO_4$:** In a dry test tube, to a little of the salt mixture a few drops of conc. $H_2SO_4$ was added. ( without heating) | White fumes evolved or Colourless pungent gas (HCl) evolved, fumed in moist air (or when blown by air from mouth) | Chloride ($Cl^-$) present |
| | Reddish brown vapours evolved | Bromide ($Br^-$) present |
| **With heating** | vinegar smell observed | Acetate present |
| | Light reddish brown vapours evolved. | Nitrate ($NO_3^-$) present |
| 4. **Test for Borate:** On a watch glass, to a little of the salt mixture the same quantity of Calcium fluoride ($CaF_2$) powder was mixed and made a paste by adding a few drops of conc. $H_2SO_4$ | Green edged flame observed. (the volatile $BF_3$ formed in the test imparted green colour to flame) | Borate ($BO_3^{3-}$ or $B_2O_7^{2-}$) present |

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| | | |
|---|---|---|
| and exposed the paste to colourless flame with the help of a glass rod. | | |
| 5. **Test for Phosphate:** A little of the salt mixture was dissolved in a minimum of conc. $HNO_3$ by heating, and excess of ammonium molybdate added. | A canary yellow precipitate, $(NH_4)_3PO_4.12MoO_3$, formed. | Phosphate ($PO_4^{3-}$) present |
| 6. **Test for Sulphate:** To the salt mixture solution barium chloride solution was added. | A white precipitate ($BaSO_4$) formed, insoluble in conc. HCl or conc. $HNO_3$ | Sulphate ($SO_4^{2-}$) present |

## CONFIRMATORY TESTS FOR ANIONS

| | | |
|---|---|---|
| **1. $BaCl_2$ test for carbonate:** To the salt mixture solution (prepared in water) $BaCl_2$ solution was added. | White precipitate ($BaCO_3$) formed, soluble in dil. HCl | Carbonate confirmed |
| **2. Ethyl or Amyl acetate test for acetate:** In a dry test tubea little of the salt mixture, a few drops of conc. $H_2SO_4$ and ethyl alcohol or amyl alcohol were taken and warmed. The contents in the test tube were poured in a beaker full of water. | A fruity smell of ethyl acetate ($CH_3COOC_2H_5$) observed. or Ripen banana smell of amyl acetate ($CH_3COOC_5H_{11}$) observed. | Acetate confirmed |
| **3. $MnO_2$ test for halides:** In a dry test tube, a little of the saltmixture and manganese dioxide were wet with a few drops of conc. $H_2SO_4$ and then heated. | Greenish yellow chlorine gas ($Cl_2$) evolved. | Chloride confirmed |
| | Reddish brown bromine gas ($Br_2$) evolved. | Bromide confirmed |

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|---|---|---|
| **4. Chromyl chloride test for chloride**:<br>In a dry test tube a little of the salt mixture and potassium dichromate were taken and wet with a few drops of conc. $H_2SO_4$ and then it was heated. | Dark red chromyl chloride vapours ($CrO_2Cl_2$) formed and passed downwards from test tube mouth. Nearby the mouth another test tube containing lead acetate solution was brought. The vapours were dissolved in the solution and yellow precipitate ($PbCrO_4$) formed. | Chloride confirmed |
| **5. Copper turnings test for nitrate:**<br>In a dry test tube a little of the salt mixture, few copper turnings and few drops of conc. $H_2SO_4$ were taken and heated. | Reddish brown vapours evolved.<br>or<br>Formation of light brown gas ($NO_2$) intensified. Mass in the test tube turned green (due to formation of anhydrous copper nitrate). | Nitrate ($NO_3^-$) present |
| **6. Ethyl borate test for borate:**<br>In a dry test tube a little of the salt mixture, few drops of conc. $H_2SO_4$ and ethyl alcohol were taken and warmed. The vapours evolved were exposed to flame and continued warming of the test tube. | The vapours were continued to burn with green edge flame (the volatile ethyl borate formed imparted green colour to the flame). | Borate confirmed |

## Soda Extract tests for further confirmation

**Preparation of sodium carbonate extract:**

The salt mixture was mixed with twice or thrice the amount of sodium carbonate. Sufficient water was added, boiled and filtered. The filtrate is known as sodium carbonate extract or **soda extract**.

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| | | |
|---|---|---|
| **1.Test for sulphate:**<br>A small portion of the soda extract was taken. On adding dil. HCl formation of bubbles started. Addition of the acid continued until the bubbles stopped (neutralisation). $BaCl_2$ solution added. | White precipitate ($BaSO_4$) formed insoluble in conc. HCl or conc. $HNO_3$. | Sulphate ($SO_4^{2-}$) confirmed |
| **2. Test for acetate**:<br>The soda extract was neutralized with dil. HCl and neutral ferric chloride solution was added. (Add $NaHCO_3$ to $FeCl_3$ solution until bubbles stopped to prepare neutral $FeCl_3$ solution) | Red precipitate, **$Fe(CH_3COO)_3$**, formed. | Acetate confirmed |
| **3. Confirmatory tests for halides:**<br>A small portion of the soda extract was neutralized with dil. $HNO_3$. Then $AgNO_3$ solution added. | White precipitate (AgCl) formed, completely soluble in ammonia solution | Chloride confirmed |
| | Pale yellow precipitate (AgBr) formed, sparingly soluble in ammonia solution | Bromide confirmed |
| **4. Brown ring test for nitrate:**<br>A small portion of the soda extract was neutralized with dil. $H_2SO_4$. Then freshly prepared $FeSO_4$ solution was added in the same quantity. To this Conc. $H_2SO_4$ was added drop by drop from sides of the test tube by keeping it in inclined position. | A brown ring, $[Fe(NO)]SO_4$, formed at the junction of the two layers. | Nitrate confirmed |

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| | | |
|---|---|---|
| **5. Test for phosphate**: A small portion of the soda extract was neutralized with dil. $HNO_3$. To this a few drops of $AgNO_3$ added and filtered. To the filtrate ammonia solution added drop by drop. | A pale yellow ring, $(NH_4)_3PO_4.12MoO_3$, formed at the junction of the two layers. | Phosphate confirmed |

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# TESTS FOR CATION

Preparation of Original Solution

The solvent at top of the list in observation column for the solubility test was taken. The given salt mixture was added to it little by little and shaken well to get a clear solution. It is O.S.

## Tests for ammonium cation ($NH_4^+$)

| Experiment | Observation | Inference |
|---|---|---|
| **1. NaOH test:** To a little of the salt mixture NaOH was solution added and warmed. | Colourless gas with ammonia smell ($NH_3$ gas) observed. A glass rod dipped in HCl solution when exposed to this gas gave dense white fumes ($NH_4Cl$). | Ammonium ion present |
| **2.Nessler's reagent test**: To the salt mixture solution excess of NaOH solution and few drops of Nessler's reagent (alkaline solution of $K_2HgI_4$) were added. | Reddish-brown (chocolate colour) precipitate or solution, $NH_2.HgO.HgI.H_2O$ (Iodide of Millon's base), formed. | Ammonium ion confirmed |

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# GROUP SEPARATION TABLE

<table>
<tr><td colspan="5">To the original solution dil.HCl is added and centrifuged</td></tr>
<tr><td rowspan="6">Residue:<br><br>$PbCl_2$ (white ppt)</td><td colspan="4">Centrifugate: $H_2S$ gas is passed through the centrifugate and centrifuged</td></tr>
<tr><td rowspan="5">Residue:<br><br>Black – CuS<br>Brown - BiS<br>Yellow - CdS</td><td colspan="3">Centrifugate: The centrifugate is boiled off till $H_2S$ is eliminated. After cooling excess of $NH_4Cl$ and $NH_4OH$ are added and centrifuged</td></tr>
<tr><td rowspan="4">Residue:<br><br>White gelatanious- $Al(OH)_3$<br>Dirty green- $Fe(OH)_2$<br>Brown- $Fe(OH)_3$</td><td colspan="2">Centrifugate: $H_2S$ gas is passed through the centrifugate and centrifuged.</td></tr>
<tr><td rowspan="3">Residue:<br><br>White - ZnS<br>Flesh - MnS<br>Black – NiS,CoS</td><td colspan="2">Centrifugate: Centrifugate is boiled off till $H_2S$ is eliminated. After cooling excess of $NH_4Cl$, $NH_4OH$ and $(NH_4)_2CO_3$ are added and centrifuged.</td></tr>
<tr><td>Residue:<br><br>White- $BaCO_3$<br>$SrCO_3$<br>$CaCO_3$</td><td>Centrifugate: After boiling, this is used in the identification of VI group cations.</td></tr>
</table>

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# I GROUP ANALYSIS

To the ppt. obtained in I group distilled water was added, boiled and filtered.

| **Residue is not formed** | **Filtrate** |
|---|---|
| | It was divided into two parts.<br>**Part I:** To this part potassium chromate solution was added. Yellow ppt. ($PbCrO_4$) formed. **Lead ion ($Pb^{2+}$) present.**<br><br>**Part II:** (Golden spangles test: To this part potassium iodide solution was added. Yellow ppt. (PbI) formed. To the separated ppt. distilled water was added, boiled, and on cooling under tap water, particles like golden yellow spangles (PbI) were appeared. **Lead ion ($Pb^{2+}$) present**. |

# II GROUP ANALYSIS

($Pb^{2+}$ and $Cu^{2+}$ of Group IIA cations present)

The ppt. obtained in II group was washed with water, 33% nitric acid (1 part conc. acid + 2 parts water) was added, heated for a minute, dil. sulphuric acid was added and filtered.

| **Residue** | | **Filtrate** | |
|---|---|---|---|
| Ammonium acetate solution was added, heated and filtered. | | Ammonia solution was added until ammonia smells, warmed and filtered. | |
| **Residue** | **Filtrate** | **Residue** | **Filtrate** |
| Mercuric mercury $Hg^{2+}$ | Acidified with dilute acetic acid and potassium chromate solution was added. Yellow ppt. ($PbCrO_4$) formed.<br><br>**Lead ion ($Pb^{2+}$) present**. | Bismuth ion $Bi^{3+}$ | .<br>To this blue colour solution acetic acid and potassium ferrocyanide solution were added.<br><br>Reddish brown ppt. (Chocolate colour ppt.) $Cu_2[Fe(CN)_6]$ formed.<br><br>**Cupric ion ($Cu^{2+}$) present.** |

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## III GROUP ANALYSIS

To the ppt. obtained in III group NaOH solution was added in excess and filtered.

| Residue | Filtrate |
|---|---|
| Dil. HCl was added to dissolve it and potassium ferrocyanide solution was added. Deep blue colour (Prussian blue) ppt. or solution, $Fe_4[Fe(CN)_6]_3$, formed.<br><br>**Iron ion ($Fe^{2+}$ or $Fe^{3+}$) present.** | Dil. HCl was added, and ammonia solution was added until ammonia smells.<br><br>White gelatinous ppt., $Al(OH)_3$, formed.<br><br>**Aluminium ion ($Al^{3+}$) present.** |

## IV GROUP ANALYSIS

To the ppt. obtained in IV group dil. HCl was added, warmed and filtered.

<table>
<tr><th>Residue</th><th colspan="2">Filtrate</th></tr>
<tr><td rowspan="2">It was dissolved in minimum of aqua regia (3 parts Conc. HCl + 1 part Conc. $HNO_3$) and was diluted by water. .<br><br>Test for Nickel:<br>To the above solution ammonia solution was added until ammonia smells. Few drops of Di Methyl Glyoxime (DMG) reagent was added. Rose coloured ppt.,<br>$[Ni(DMG)_2]$ complex, formed.<br><br>Nickel ion, $Ni^{2+}$ present.</td><td colspan="2">It was boiled to expel $H_2S$ gas. NaOH solution was added in excess, boiled and filtered.</td></tr>
<tr><td>Residue</td><td>Filtrate</td></tr>
<tr><td></td><td>Conc.$HNO_3$ was added to dissolve the residue, lead dioxide was added, boiled, cooled and diluted with water. Purple pink colour, $Mn(OH)_2$, formed.<br><br>Manganese ion ($Mn^{2+}$) present.</td><td>$H_2S$ was passed. White ppt. (ZnS) formed.<br><br>Zinc ion ($Zn^{2+}$) present.</td></tr>
</table>

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## V GROUP ANALYSIS

The ppt. obtained in V group was dissolved in dil. acetic acid and was divided into three parts.

| Part I | Part II | Part III |
|---|---|---|
| Potassium chromate solution was added. Yellow ppt. ($BaCrO_4$) formed.<br><br>**Barium ion ($Ba^{2+}$) present** | Ammonia solution was added until ammonia smells, and ammonium sulphate solution was added and shaken well. White ppt. ($SrSO_4$) formed.<br><br>**Strontium ion ($Sr^{2+}$) present**. | Ammonia solution was added until ammonia smells, and ammonium oxalate solution was added. White ppt. ($CaC_2O_4$) formed.<br><br>**Calcium ion ($Ca^{2+}$) present.** |

## VI GROUP ANALYSIS

Before going to the General Group Table ammonium ion was tested. So, tests were done for the remaining cation in this VI group potassium cation ($K^+$) only.

| Experiment | Observation | Inference |
|---|---|---|
| 1.To the O.S. acetic acid and sodium cobaltinitrite solution were added. | Yellow ppt. $K_3[Co(NO_2)_6]$ formed. | Potassium ion ($K^+$) present. |
| 2.To the O.S. tartaric acid solution was added and sides of the test tube were scratched with a glass rod. | White ppt., $(COOH)(CHOH)_2(COOK)$, formed | Potassium ion ($K^+$) present. |

**Report:** The given salt mixture contains the following ions.

**Anions: .....**

**Cations: .....**

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