Chapter 1

SEAT (Student, Environment, Administrator, Teacher)
Friendly Microscale Chemistry Laboratory

1.1 Introduction

The crucial role of practical work and experimentation in science curriculum is universally accepted. This is more so in chemistry which is an experimental science. Most of the concepts in this area are better understood by doing the practical work.

The aims and objectives of practical work in the laboratory can be stated as follows:

1. better understanding of scientific concepts and principles.
2. promotion of basic skills and competencies (procedural and manipulative skills, observational skills, drawing skills, reporting and interpretation skills).
3. awakening and maintaining curiosity in the learning environment.

The laboratory work is an indispensable part of chemistry instruction. Despite several laudable efforts in the past, experiments, by and large, have continued to be marginalised in school education, and have not received the due importance. There are several factors which contribute towards neglect of the laboratory work. In general, there is a crunch of financial resources for constructing a separate conventional chemistry laboratory. Increasing cost of chemicals, handling a large number of students, no time to maintain equipment and accessories, no money to replace the breakages, problems in disposal of waste, handling of toxic chemicals, working in shifts are other major contributing factors.

In order to overcome the above problems, the desired modifications may be

- change in facilities.
- modifications in laboratory programmes.
- optimisation in the use of resources.
- adaptation of cost-effective, safe and efficient techniques.
- a move towards “green chemistry”.
Alternative ideas to replace the conventional laboratory while meeting the objectives of practical work are being thought of. Recent developments include microscale chemistry experiments using small quantity of chemicals and simple equipment. The concept was first introduced by D.W. Mayo, S.S. Butcher et al. at Bowdoin College in Brunswick about 30 years ago. In India, this technique has been developed and introduced in the school system by Prof. H.O. Gupta, NCERT with some innovative laboratory apparatus known as microscale chemistry laboratory kit.

1.2 What is Microscale Chemistry Laboratory Kit?
This microscale chemistry laboratory kit enables the students to perform experiments in an environmentally safe pollution free atmosphere using small quantities of chemicals without compromising the quality and standard of experiments. Apart from the above, we have designed and developed a concept of microscale laboratory where the students need not wander around in search of reagents. The experiments can be performed quickly. They are thus, pollution and hazard free. Conventional laboratory racks and bottles are replaced by a small box containing all the small labwares and apparatus. On the top of the box are revolving circular racks to hold plastic dispenser bottles which dispense one drop of liquid at a time. In short, this method can be called Student, Environment, Administrator, and Teacher (SEAT) friendly.

1.3 How is it SEAT friendly?
Students can perform the experiments easily.

Student Friendly
- Our laboratory kit uses solid chemicals in few milligrams quantities and liquid chemicals in few drops using special low-cost apparatus instead of grams and full test tube chemicals as in traditional laboratories.
- The chemicals used are in very small quantity, thereby reducing fumes and the risks of accidents, acid burns, etc.
- The experiments are quick to perform, thus, saving time for performing more experiments.
- It develops habit of conservation.
**Environment Friendly**

This kit
- Reduces use of chemicals promoting waste reduction at the source.
- Offers vastly improved laboratory safely by better laboratory air quality through reduction in:
  - exposure to toxic chemicals
  - fire and explosion hazards
  - spills and accidents
- Provides clean and congenial environment

**Administrator Friendly**
- It sharply reduces laboratory cost.
- It lowers glass breakage cost.
- It saves storage space.

**Teacher Friendly**
- It promotes better student-discipline in the laboratory.
- It is pedagogically superior. Teachers can ask the students to do more experiments during the saved time to help in better conceptual understanding.

1.4 What new apparatus are provided in the kit?

Below is the list of some of the apparatus provided.

1. Small portable box with revolving top for easy access to chemicals and apparatus, replacing big racks.
2. Polyethylene dispensing bottles (squeeze type) to dispense liquid chemicals drop wise avoiding contamination of chemicals.
3. Well plates for fast and easy precipitation.
5. Aluminum block for safe determination of melting point and boiling point.
6. Kerosene burner
7. Micro burettes.
8. Plastic droppers for easy transfer of liquids.
9. Miniature glasswares for least consumption of chemicals.
10. Micro filtration unit.
11. Micro spatulas

1.5 **How many Chemistry experiments can be done with this kit?**

All the experiments at school level from Class VI to XII can be performed using this kit. We mention a few examples below:

**Microscale Experiments in Analytical Chemistry**
- Microscale Titration
- Microscale Gravimetric Determination
- Microscale Qualitative Analysis.

**Microscale Experiments in Physical Chemistry**
- Potentiometric titrations.
- Determination of solubility product.
- Verification of Nernst’s equation
- Determination of $pK$ of an acid.
- Determination of equilibrium, constant, pH and all other physical chemistry experiments.

**Microscale Experiments in Organic Chemistry**
- Organic qualitative analysis
- Small-scale distillation and preparation.
- Tests for functional groups.

A model micro scale chemistry laboratory has been set up in NCERT after tryout in some selected schools of Delhi. A book on microscale chemistry experiments has also been published and the required micro apparatus have also been developed. It may be mentioned that microscale technique is being followed in many parts of the world for the last twenty-five years or so in the form of kits for individual experiments, especially in organic chemistry, but we have designed and developed a laboratory in the form of kit comprising some novel apparatus enabling the students to perform all the chemistry experiments at school level fostering discipline and habit of
conservation in them. It is truly cost-effective, interesting and safe way of performing chemistry experiments. Instead of using big racks to store chemicals, all necessary chemicals and miniature apparatus are kept in a specially designed box with revolving top. Students do not even need to move from their place to perform the experiments. W-tube which the teachers call wonder tube makes the gas absorption tests fast, safe and almost smell free. Even the lingo differs. We do not talk of pouring or adding chemicals from one containers to another, as they imply quantity. We talk of transferring them.

1.6 Comparison of Microscale Chemistry Laboratory Kit and Traditional Chemistry Laboratory

<table>
<thead>
<tr>
<th>Traditional Chemistry Laboratory</th>
<th>Microscale Chemistry Laboratory</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. <strong>Quantity of Chemicals Used</strong>&lt;br&gt;A few grams of solid and a few mL of solutions are used.</td>
<td>A few milligrams and drops of solutions are used. A typical microscale experiment will consume only, say less than 1% of the required quantity of reagents used in a corresponding traditional macroscopic experiment.</td>
</tr>
<tr>
<td>2. <strong>Environment Protection</strong>&lt;br&gt;In a traditional chemistry laboratory, lot of chemicals are drained. Lots of toxic fumes cause air pollution.</td>
<td>In microscale chemistry lab, use of sink may not be necessary and as such drainage of chemicals is minimised. Toxic fumes are almost non-existent.</td>
</tr>
<tr>
<td>3. <strong>Accidental Hazards</strong>&lt;br&gt;Many accidents occur due to fire, acid burns etc.</td>
<td>Flame intensity is minimised and acid burns are almost non-existent. Risks of fire hazards and acid burns are reduced to minimum.</td>
</tr>
<tr>
<td>4. <strong>Breakage of Glassware</strong>&lt;br&gt;Lot of breakage of glassware.</td>
<td>Breakage is minimised.</td>
</tr>
<tr>
<td>5. <strong>Time taken for an Experiment</strong>&lt;br&gt;Lot of time is required for a particular experiment.</td>
<td>Experiments are very fast. So more experiments can be done in the same allotted time.</td>
</tr>
<tr>
<td>6. <strong>Cost</strong>&lt;br&gt;A typical traditional laboratory cost about 4-5 lakhs to set up initially and recurring cost of Rs. 50,000/- per year for consumable like glassware chemicals for a batch of 24 students.</td>
<td>There is lot of cost reduction. Each kit is suitable for four students and can be managed within Rs. 30,000/- at the initial cost for a batch of 24 students. Recurring cost of chemicals and glassware is minimum. Chemicals once purchased would last for many years.</td>
</tr>
</tbody>
</table>
1.7 Summary

Use of microscale laboratory techniques has many benefits:

- The chemical waste generation in the schools is reduced markedly.
- Students learn waste minimisation techniques that they will ultimately practise throughout their lives.
- Schools will continue to offer hands-on laboratory experience to their students, a practice that has been threatened by the increasing costs and difficulty of waste disposal, it will introduce laboratory work even in the institutions which are too poorly equipped.
- Health risks to students and teachers in the school laboratories are minimised.
- The cost of materials and equipment needed to provide students with an excellent science education is reduced to a significant extent.
- Educational laboratories are safer environmentally for both students and teachers when microscale methods are used.
- Saves time for preparation.
- Reduces waste at the source.
- Ensures more safety.
- Lowers the costs for chemical substances and equipment.
- Requires smaller storage area.
- Reduces reliance on intensive ventilation systems with a significant reduction in electricity consumption.
- Provides pleasant working atmosphere.
- Decreases reaction time.
## Chapter 2

### 2.1 Description of Kit Items

<table>
<thead>
<tr>
<th>S.No</th>
<th>Item Name (Quantity/kit)</th>
<th>Figure/ Setup</th>
<th>Uses</th>
</tr>
</thead>
</table>
| 1.   | Wooden box with revolving top (1) | ![Wooden box](image)
|      |                          |               | 1. Storage box opens on both sides. Contains necessary apparatus for doing experiments. 2. Revolving top containing dispensing bottles and vials for easy access to chemicals. |
| 2.   | Microbeaker (10 mL) (12) | ![Microbeaker](image)
|      |                          |               | 1. As a container for keeping liquids. |
| 3.   | Beaker (50 mL) (3) | ![Beaker](image)
|      |                          |               | 1. As a container for use in electrochemical experiments, thermochemistry experiments preparation of salts, etc. |
| 4.   | Microfiltration unit (2) | ![Microfiltration unit](image) | **Steps for use**
|      |                          |               | 1. Bore the rubber cork to fit the stem of Hirsch funnel.
|      |                          |               | 2. Cut the filter paper to fit the mesh of the funnel. The size should exactly fit the mesh, neither big nor small.
|      |                          |               | 3. Fit the cork with funnel in the mouth of the boiling tube (having a side tube).
|      |                          |               | 4. Transfer the solution to be filtrated to the funnel.
|      |                          |               | 5. Press the bulb with hand to evacuate it and place it on the side tube of the boiling tube while keeping it pressed.
<p>|      |                          |               | 6. Release the pressure. The bulb will suck the air inside the boiling tube allowing fast filtration. |</p>
<table>
<thead>
<tr>
<th>S.No.</th>
<th>Item Name</th>
<th>Figure/ Setup</th>
<th>Uses</th>
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<tbody>
<tr>
<td>5.</td>
<td>Watch glass</td>
<td>(2)</td>
<td>1. The watch glass is used to hold solids when being weighed.</td>
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<td></td>
<td></td>
<td></td>
<td>2. It should never be heated.</td>
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<td>6.</td>
<td>Micro test plate (Well plate) (4)</td>
<td>1. Flat plate with multiple “wells” used as micro test tubes.</td>
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<td></td>
<td></td>
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<td>2. Contains 96 rectangular matrix.</td>
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<td>3. Each well of a micro plate typically holds somewhere between a few to a few hundred microlitres of liquid.</td>
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<td></td>
<td>4. Use these wells by placing a few drops of test solution and reagent.</td>
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<td></td>
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<td></td>
<td>5. Clean the wells with jet of water from wash bottle and dry it with cotton swab.</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>6. A number of tests can be done without bothering to clean the wells after one test only. But don’t forget to clean the wells at the end of the experiment.</td>
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<td></td>
<td></td>
<td></td>
<td>7. Do not heat the micro test plate.</td>
</tr>
<tr>
<td>7.</td>
<td>Micro measuring cylinder (2)</td>
<td>1. To measure the volume of a liquid up to 10 mL.</td>
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<td></td>
<td></td>
<td></td>
<td>2. To conduct activities requiring measurement of rise or fall in the level of a liquid.</td>
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<td>S.No.</td>
<td>Item Name</td>
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<tr>
<td>8.</td>
<td>Micro burette</td>
<td><img src="image" alt="Micro burette" /></td>
<td>1. The micro burette has 5 mL capacity with the least count of 0.2 mL. At the narrow end of the burette, a microtip is attached with the help of plastic tube. Microtip delivers about 78 drops in 1 mL of solution. 2. Two burettes are used to avoid the use of a pipette and are fixed in the stand. 3. Pour the liquid slowly and make sure that no air bubble remains inside the burettes.</td>
</tr>
<tr>
<td>9.</td>
<td>Petri dish</td>
<td><img src="image" alt="Petri dish" /></td>
<td>1. A shallow glass round dish used for chromatography.</td>
</tr>
<tr>
<td>10.</td>
<td>Micro test tubes</td>
<td><img src="image" alt="Micro test tubes" /></td>
<td>1. As a container for keeping and testing small quantities of liquids. 2. To boil water over the micro burner.</td>
</tr>
<tr>
<td>11.</td>
<td>Pasteur pipette</td>
<td><img src="image" alt="Pasteur pipette" /></td>
<td>1. Made of plastic and also known as dropper. 2. Used to transfer small amounts of liquids.</td>
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<td>S.No.</td>
<td>Item Name</td>
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<td></td>
<td>(2)</td>
<td></td>
<td>2. Used to pour liquid on to the filter paper during filtration.</td>
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<td>(4)</td>
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<tr>
<td></td>
<td>(1 box)</td>
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<tr>
<td>15.</td>
<td>Thermometer</td>
<td></td>
<td>1. Thermometer (0°C to 250°C) with 1°C least count for boiling point</td>
</tr>
<tr>
<td></td>
<td>(1 each)</td>
<td></td>
<td>and melting point determination.</td>
</tr>
<tr>
<td></td>
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<td></td>
<td>2. Thermometer (0°C-60°C) (Least count 0.1°C) for thermochemistry</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>experiments</td>
</tr>
<tr>
<td>16.</td>
<td>Stirrer</td>
<td></td>
<td>1. Circular end is used to stir a mixture or a solution in the</td>
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<tr>
<td></td>
<td>(2)</td>
<td></td>
<td>calorimeter.</td>
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<td></td>
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<td>2. The mounted stirrer is inserted in the foam lid of the</td>
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<td></td>
<td>calorimeter surrounding the thermometer.</td>
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<td>S.No.</td>
<td>Item Name</td>
<td>Figure/Setup</td>
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</tr>
<tr>
<td>17.</td>
<td>Calorimeter</td>
<td><img src="image1" alt="Image" /></td>
<td>1. Device used for measuring the enthalpy of chemical reactions or physical changes. &lt;br&gt;2. A 50 mL beaker is fixed in the foam container with the lid.</td>
</tr>
<tr>
<td>18.</td>
<td>Circular Whatman paper</td>
<td><img src="image2" alt="Image" /></td>
<td>1. Used for filtering the substances or product of a chemical reaction or in chromatography, etc.</td>
</tr>
<tr>
<td>19.</td>
<td>Kerosene burner</td>
<td><img src="image3" alt="Image" /></td>
<td>1. Used for heating and exposing items to flame. &lt;br&gt;2. The device safely burns a continuous stream of kerosene/gas. &lt;br&gt;3. To use kerosene burner, fix the lid containing wicks to the container having kerosene oil. Fix inner chimney to the lid. Light the burner. Fix outer chimney to the lid. A blue flame will appear after a few minutes.</td>
</tr>
<tr>
<td>20.</td>
<td>Tripod</td>
<td><img src="image4" alt="Image" /></td>
<td>1. Used for keeping the beaker, test tubes or chaina dish in position for heating (No wire gauze is put over the tripod stand).</td>
</tr>
<tr>
<td>S.No.</td>
<td>Item Name</td>
<td>Figure/ Setup</td>
<td>Uses</td>
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</tbody>
</table>
|       | **W-Tube** (10) | ![W-Tube Diagram](image) | 1. In one of the outer sides of W-tube, a few mg of solid or few drops of liquid under test are transferred. In the other arm, transfer a few drops of reagent (e.g. KMnO₄ solution) that would react with the gas generated in the first arm. The reagent that would react with the test sample is then transferred as one or two drops with the help of another dropper into the first arm. The stem of the dropper would make this side airtight.  
2. The generated gas would bubble through the liquid reagent in the other side showing the chemical change.  
3. Used in the reactions such as to test the and evolution of CO₂ and of SO₂ gases and in the test of ethylene.  
4. Cleaning of W-tube is done by passing a stream of water from the wash bottle jet into one of the arms of the W-tube. The second arm should point towards the container in which the contents are to be drained. |
| 22.   | **Multimeter** (1) | ![Multimeter Diagram](image) | 1. To check the continuity of a wire and circuit.  
2. To measure resistance, voltage, and current. |
<table>
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<tbody>
<tr>
<td>23.</td>
<td>Volumetric flask (2)</td>
<td><img src="image" alt="Volumetric flask" /></td>
<td>1. Used to make accurate solution by measuring a specific volume.</td>
</tr>
</tbody>
</table>
| 24.   | Dropper with rubber bulb (3)     | ![Dropper with rubber bulb](image)                                           | 1. The dropper is used for transferring small amounts of a liquid from container to another.  
2. Fix the rubber bulb to the small glass dropper. |
| 25.   | Salt bridge (2)                  | ![Salt bridge](image)                                                        | 1. Consists of U-shaped glass tubes with upper side tube in the middle, filled with a relatively inert electrolyte, usually potassium chloride or sodium chloride.  
2. Fix the bigger rubber bulb (same as used with filtration unit) or syringe to the upper side tube of U-tube.  
3. Squeeze the bulb and dip the two ends of U-tube in the saturated solution of KCl. Release the pressure. The liquid will be filled in the U-tube.  
4. Allows the flow of ions to maintain a balance in charge between the oxidation and reduction vessels while keeping the contents of each separate. With the charge difference balanced, electrons can flow once again, and the reduction and oxidation reactions can proceed. |
<table>
<thead>
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<th>Uses</th>
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</thead>
</table>
| 26.   | Dispensing bottles (30)    |               | 1. Used to keep the chemical reagents.  
       |                             |               | 2. Open the cap and squeeze the bottle to dispense one or two drops.  
       |                             |               | 3. Label the dispensing bottle.                                               |
| 27.   | Vials (20)                 |               | 1. Used as a container to store the solid chemicals.                                      |
| 28.   | Round bottom flask (2)     |               | 1. Round-bottom flask is used in a variety of applications where the contents are heated or boiled in organic preparations. |
| 29.   | China dish (2)             |               | 1. Used to heat small quantities to very high temperatures.  
<pre><code>   |                             |               | 2. Used as a water bath.                                                        |
</code></pre>
<p>| 30.   | Fusion tube (20)           |               | 1. To determine the boiling point of a substance.                                          |</p>
<table>
<thead>
<tr>
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<tbody>
<tr>
<td>31.</td>
<td>Chromatography jar with cork</td>
<td></td>
<td>1. Put the cork on the open end of the jar, fit a hook in the inner side of the bottom of the cork so as to hang the Whatman paper strip. <strong>Note:</strong> Strip width should be of ( \frac{3}{4} ) of inch, the length must be shorter than that of the jar, so that it would not touch the inner walls of the jar.</td>
</tr>
<tr>
<td></td>
<td>(1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>32.</td>
<td>Wash bottles</td>
<td></td>
<td>1. Used to contain water to wash the apparatus like micro test plate, W-tube, micro test tube etc.</td>
</tr>
<tr>
<td></td>
<td>(2)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>33.</td>
<td>Micro spatula</td>
<td></td>
<td>1. Used for transferring small amounts of solid</td>
</tr>
<tr>
<td></td>
<td>(2)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>34.</td>
<td>pH paper (a pack)</td>
<td></td>
<td>1. Used to determine the pH or to study the change in pH of the chemical reaction.</td>
</tr>
</tbody>
</table>
| 35.   | Aluminium block                  |              | 1. Used as a platform to heat the test tubes.  
2. Used to hold the thermometer and measure the temperature.  
3. The narrow slit is used to place the capillary tube for melting point determination.  
4. Put the thermometer in one of the holes.  
5. Put the fusion tube containing a liquid when boiling point is to be determined. Make sure the level of the liquid should be visible. Place an inverted capillary tube sealed at one end. |
<p>|       | (1)                             |              |                                                                                                                                 |</p>
<table>
<thead>
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<tbody>
<tr>
<td>36.</td>
<td>Forceps</td>
<td></td>
<td>1. Used to pick and hold the objects.</td>
</tr>
<tr>
<td>37.</td>
<td>Two way burette clamp and stand (2)</td>
<td></td>
<td>1. Used to hold the burette upright during the titration.</td>
</tr>
<tr>
<td>38.</td>
<td>Micro test tube holder (2)</td>
<td></td>
<td>1. The holder is used to hold test tubes when they are hot and untouchable.</td>
</tr>
<tr>
<td>39.</td>
<td>Electrodes (1 pair)</td>
<td></td>
<td>1. Used to generate voltage in an electrochemical reaction.</td>
</tr>
<tr>
<td>40.</td>
<td>Micro test tube brush (2)</td>
<td></td>
<td>1. The test tube brush is used to easily clean the inside of a test tube.</td>
</tr>
<tr>
<td>41.</td>
<td>Micro test tube rack (2)</td>
<td></td>
<td>1. The test tube rack is used to hold test tubes.</td>
</tr>
<tr>
<td>42.</td>
<td>Micro funnel (2)</td>
<td></td>
<td>1. Used for filtering.</td>
</tr>
</tbody>
</table>
Chapter 3

DESCRIPTION OF EXPERIMENTS

3.1 CLASS XI

Characterisation and purification of chemical substances

Experiment 1: (a) To determine of boiling point of an organic compound

Apparatus required
Aluminium block, capillary tube, fusion tube, thermometer, stand (with clamp), tripod, and kerosene burner.

Chemicals required:
Organic liquids such as: Benzene, Benzaldehyde for boiling point determination.

Procedure
(1) Take the Aluminium block. Light the kerosene burner.
(2) Take a capillary tube and close its one end by holding the end in the flame for 2-3 minutes.
(3) Transfer a few mL of the organic liquid compound in the fusion -tube. Dip the capillary tube into the liquid in the fusion tube keeping the sealed end up.
(4) Put the tube in one of the holes of aluminium block and put the thermometer in

Fig.1.1: Arrangement showing boiling point determination
the other hole. Care should be taken that the liquid is visible in
the fusion tube after it is put in the hole.

(5) Place the Aluminium block on the tripod.
(6) Start heating the block with the help of kerosene burner.
(7) Note the temperature as soon as regular streams of bubbles are
seen out of liquid in the fusion tube.

**Observation**

1. Boiling point of water = 100°C
2. Boiling point of benzene = 78°C
3. Boiling point of Benzaldehyde = 178°C
Experiment 2: To determine of melting point of an organic compound.

Apparatus required
Aluminium block, capillary tube, thermometer, stand (with clamp), tripod and kerosene burner

Chemicals required
Organic compound such as Naphthalene and benzoic acid for melting point determination

Procedure
1. Take a capillary tube and close its one end by heating the end in the flame for 2-3 minutes while continuously rotating it.
2. Take the given organic compound say naphthalene, on a tile and grind it into a very fine powder.
3. Hold the closed end of the capillary tube between your thumb and finger. Now dip its open end in the fine powder of the organic compound. Gently tap the capillary tube on the table to fill the compound in capillary tube to a length of about 1–2 cm. Place capillary tube in the groove of aluminium block. Make sure that portion of the capillary tube containing the organic substance is in the middle of the groove.
4. Put the thermometer in one of the holes.
5. Place the Aluminium block on tripod above the kerosene burner and start heating the block with the burner.
6. Note the temperature as soon as the substance begins to melt.
Purification of Compounds by Crystallisation

**Experiment 3: To crystallise the pure compound from an impure sample of anyone of the following-Alum, Copper sulphate, Benzoic acid**

**Apparatus required**
Beaker( 50 mL), watch glass, filtration unit and kerosene burner.

**Preparation of crystals of CuSO₄·5H₂O**

**Chemicals required**
CuSO₄·5H₂O and dil. H₂SO₄

**Procedure**

1. Prepare a clear solution of CuSO₄·5H₂O by dissolving its 0.8 g in about 8 mL of water. Add 1-2 drops of 6 mL H₂SO₄ to it.
2. Heat the solution for a while and filter it using filtration unit.
3. Transfer the solution from filtration unit to another beaker. Allow it to stand come to room temperature. The crystals will appear.
4. Filter, wash and dry the crystals.
5. Weigh the crystals and report the yield.

Crystallisation of Alum, benzoic acid and iron sulphate can also be done by following the above procedure. The amount of substances required is as follows:

- For CuSO₄·5H₂O-
  - 0.8 g CuSO₄+1-2 drops dil. H₂SO₄ + 8 mL H₂O
- For FeSO₄·7H₂O-
  - 0.5g sample of FeSO₄ + 3 drops of H₂SO₄ + 3mL H₂O
- For Alum-
  - 1.0 Alum sample +2 drops of H₂SO₄ + 5 mL H₂O
- For Benzoic acid-
  - 0.5 g of Benzoic acid + (8 mL H₂O)
Chemical Equilibrium

Experiment 4: To study the pH change by common ion effect in case of weak acids and weak bases.

Apparatus required
Well plates and dropper/Pasteur pipette

Chemicals required
NH₄OH, NH₄Cl and Universal indicator solution / paper

Fig.4.1: Testing in a well plate

Observation table for NH₄OH and NH₄Cl

Table I

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Sample solution</th>
<th>Colour produced</th>
<th>pH value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>3 drops NH₄OH+3 drops of H₂O</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>2.</td>
<td>3 drops NH₄OH+3 drops of NH₄Cl</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3.</td>
<td>3 drops NH₄OH + 1 drops of NH₄Cl</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>4.</td>
<td>3 drops NH₄OH + 30 drops of NH₄Cl</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>5.</td>
<td>30 drops NH₄OH + 3 drops of NH₄Cl</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
Comments

It may be performed in micro test tubes by counting no. of drops being added.

Observation Table for common ion effect (CH$_3$COOH and CH$_3$COONa)

<table>
<thead>
<tr>
<th>S.No</th>
<th>Sample solution</th>
<th>Colour produced</th>
<th>pH value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>1 mL CH$_3$COOH</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>2.</td>
<td>1 mL CH$_3$COOH +0.1 g CH$_3$COONa</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3.</td>
<td>1 mL CH$_3$COOH + 0.2 g CH$_3$COONa</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>4.</td>
<td>1 mL CH$_3$COOH + 0.3 g CH$_3$COONa</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Table III (NH$_4$OH and NH$_4$Cl)

<table>
<thead>
<tr>
<th>S.No</th>
<th>Sample solution</th>
<th>Colour produced</th>
<th>pH value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>1 mL NH$_4$OH+ 0.05g NH$_4$Cl</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>2.</td>
<td>1 mL NH$_4$OH+ 0.10 g NH$_4$Cl</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3.</td>
<td>1 mL NH$_4$OH+ 0.15 NH$_4$Cl</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>4.</td>
<td>1 ml NH$_4$OH+ 0.20 NH$_4$Cl</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
Experiment 5: To study the shift in equilibrium between the chemical systems by increasing the concentration of either ions or the temperature.

Apparatus
Glass rod, micro test tubes, spatula, test tube stand, micro beakers, droppers, well plate, Pasteur pipette and kerosene burner.

Chemicals required:
2% FeCl$_3$, 2% NH$_4$SCN, CoCl$_2$ or Cu(NO$_3$)$_2$, 1 M HCl and conc. HCl

Procedure:
(a) Experiment with FeCl$_3$ and NH$_4$SCN

Effect of Change in Concentration
1. Take 6 mL of water in a micro beaker and add 2 drops of ferric chloride solution and 2 drops of NH$_4$SCN. It is an almost colourless solution with light yellow tinge. Record the colour of the solution. Transfer 2 drops of this solution into 3 wells of well plate.
2. To well-1, add 2 drops of water and use it to control for comparison in next steps.
3. Increase the conc. of FeCl$_3$ in well-2 by adding 2 drops of FeCl$_3$ solution. Mix, observe and record whether the solution is less or more red or is of the same colour as in the well-1.
4. Add 2 drops of NH$_4$SCN solution in the well-3. Mix, observe and record the colour of solution as compared to the solution in well-1.

Fig 5.1 Showing the use of different apparatus
Effect of Change in Temperature

1. Pass the glass rod through the flame of the kerosene burner three or four times. Put the rod in the well-2. Describe the colour change.

2. Remove the rod, clean it with water and dip in cold water and insert in well plate-2. Describe the colour change.

(b) Experiment with CoCl₂ and Cl⁻ ions

1. Place 1 spatula of solid cobalt chloride into each of the two micro beakers / test tubes.

2. One test tube is kept as standard for comparison.

3. Using a dropper, add 6 drops of water into both test tubes-1 and 2. Use the tip of spatula to stir until all of the crystals have dissolved. Note the colour of the solution.


5. Wait for about 30 seconds, then add 5-6 drops of water in test tube-2. Note the colour.
Volumetry

Experiment 6: To prepare a standard solution of (0.1M) oxalic acid. Apparatus required

Measuring flask 25 mL, funnel, glazed paper/aluminium foil, wash bottle, balance with fractional weight box and spatula

Chemical Required

Oxalic acid

Procedure

1. Weigh an empty, clean and dry glazed paper/butter paper/aluminum foil accurately.
2. Weigh 0.315 g oxalic acid on the glazed paper for making 25 mL solution in measuring flask.
3. Transfer the oxalic acid carefully from the paper into the clean and dry measuring flask. Wash the glazed paper and funnel several times with distilled water. While washing the funnel, water should be added in small amount so that volume does not exceed ¼ of the volume of the measuring flask.
4. Swirl the measuring flask till the solid oxalic acid completely dissolves and make the volume up to the etched mark on the measuring flask with the distilled water and shake it.
Experiment 7: To determine the strength of a given solution of sodium hydroxide by titrating it against the standard solution of oxalic acid

Apparatus required

5 mL burette-2, flasks-2, two-way clamp with standard measuring flasks (25 mL) -2

Chemicals

0.05 M oxalic acid, 0.05 M sodium hydroxide (approximate) and phenolphthalein

Procedure

A.

1. Clean the burettes thoroughly, wash with distilled water and finally rinse one burette with sodium hydroxide solution and other with oxalic acid solution. Fix them in the stand with the help of two-way clamp.
2. Fill one burette with the sodium hydroxide solution through the syringe up to the zero level.
3. Remove the air gap if any from the burette by running the solution forcefully.
4. Note the initial reading by keeping the eye exactly at the level of the meniscus of the solution. Always remember that for all the colourless solutions in the burette. The reading coinciding with lower meniscus is noted and for the coloured solutions, the reading coinciding with upper meniscus is noted.

B

1. Fill the other burette with oxalic acid solution.
2. Add 1 mL of oxalic acid and 1-2
drops of phenolphthalein indicator to the filtration flask, place it over the glazed tile and titrate the acid with sodium hydroxide solution till a very faint permanent pink colour is obtained. Don’t throw the solution. To this solution add 0.5 mL oxalic acid solution and again titrate it with NaOH solution. Take 4 or 5 readings by adding oxalic acid solution in increment of 0.5 mL and titrating it with NaOH.

**Observation Table:**

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Oxalic acid ( burette A) $V_1$</th>
<th>NaOH solution ( burette B) $V_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.0</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>1.5</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>2.0</td>
<td>-</td>
</tr>
<tr>
<td>4</td>
<td>2.5</td>
<td>-</td>
</tr>
</tbody>
</table>

**Calculation:**

\[
A_1 M_1 V_1 = A_2 M_2 V_2
\]

(Oxalic Acid) (Sodium hydroxide)

Calculation of Strength = Molarity x molecular mass
Experiment 8: To prepare the standard solution of \((0.1\text{M})\text{Na}_2\text{CO}_3\)

Apparatus required:
25 mL measuring flask, glazed paper, funnel, wash bottle, balance with weight box

Chemicals required:
\(\text{Na}_2\text{CO}_3\)

Procedure:
1. Weigh a clean and dry glaze paper /butter paper /aluminum foil accurately.
2. Weigh 0.26 g sodium carbonate on the glaze paper for making solution in 25 mL measuring flask.
3. Transfer the sodium carbonate carefully from the paper into the clean and dry measuring flask. Wash the glaze paper and funnel several times with distilled water. While washing the funnel, water should be added in small amount so that it does not exceed \(1/4\text{th}\) of the volume of the measuring flask.
4. Swirl the measuring flask till the solid sodium carbonate completely dissolves. Make the volume up to the etched mark on the measuring flask with the distilled water and shake it uniformly.
Experiment 9: To determine strength of a given solution of hydrochloric acid by titrating it against standard Na₂CO₃ solution.

Apparatus required
5 mL burette-2, 10 mL beaker or titrating flask and two-way clamp with stand.

Chemical required
0.1M Na₂CO₃, dil. HCl solution and methyl orange solution

Procedure—same as in experiment No. 7
In this case, hydrochloric acid should be taken in the burette B and sodium carbonate solution in the burette A and methyl orange should be used as an indicator. The colour change at the end point will be from yellow to pink.

Fig.9.1: Arrangement showing two-burette method
### Observations

<table>
<thead>
<tr>
<th>S.No.</th>
<th>( \text{Na}_2\text{CO}_3 \text{ solution ( burette A)} )</th>
<th>( V_1 )</th>
<th>( \text{dil. HCl solution ( burette B)} )</th>
<th>( V_2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td>1.0</td>
<td></td>
<td>—</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>1.5</td>
<td></td>
<td>—</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td>2.0</td>
<td></td>
<td>—</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td>2.5</td>
<td></td>
<td>—</td>
</tr>
</tbody>
</table>

### Calculations

Using morality equation

\[
A_1 \ M_1 \ V_1 = A_2 \ M_2 \ V_2
\]

\( A_1 \) (\( \text{Na}_2\text{CO}_3 \)) = 2

\( A_2 \) (\( \text{HCl} \)) = 1

Calculate \( M_2 \)

**Strength** = \( M_2 \times 36.5 \text{ g/L} \)
Qualitative analysis

Experiment 10: To detect one cation and one anion from the following:

Cations - Pb²⁺, Cu²⁺, As³⁺, Al³⁺, Fe³⁺, Mn²⁺, Ni²⁺, Zn²⁺, Co²⁺
Ca²⁺, Sr²⁺, Ba²⁺, Mg²⁺, NH₄⁺
Anions - CO₃²⁻, S²⁻, SO₃²⁻, SO₄²⁻, NO₂⁻, NO₃⁻, Cl⁻, Br⁻, I⁻, PO₄³⁻, CH₃COO⁻
(Insoluble salts to be excluded)

Apparatus:
Micro test-tubes with stand, test tube holder, kerosene burner, W-tube, well plate, dispensing bottles, china dish, cotton swabs, charcoal cavity, W-Tube etc.

Chemicals:
6MH₂SO₄, 6MHCl, lime water, sodium carbonate, sodium metal, BaCl₂ solution, KMnO₄ solution, KI solution, starch solution, acetic acid, conc. H₂SO₄, NH₄OH, MnO₂, Cu turnings, AgNO₃ solution, K₂Cr₂O₇, lead acetate solution, CCl₄, ethanol, ammonium molybdate, conc HNO₃, borax, cobalt nitrate, Nessler’s reagent, bromine water, chlorine water, lime water, etc.

Introduction:
The inorganic qualitative analysis involves the identification of ions present in a sample. The salt consists of anion and cation. The basis of identification is the observed chemical behaviour of the components. The salt analysis may be systematically carried out in the following three steps:

i) Preliminary examination
ii) Detection of anions and
iii) Detection of cations

Preliminary examination:
First we proceed to examine the physical properties of the salt. This is followed by the action of heat on the salts and also certain tests, such as flame test etc.

Detection of Anions:
A preliminary hint about some anions may be obtained by the formation of volatile or gaseous products when the salt is treated with
i) dil. H$_2$SO$_4$ (for CO$_3^{2-}$, S$^{2-}$, SO$_3^{2-}$, NO$_2^-$ and CH$_3$COO$^-$)

ii) conc. H$_2$SO$_4$ (for Cl$^-$, Br$^-$, I$^-$ and NO$_3^-$)

1. Take about 10 mg of the salt in a dry test tube with the help of a spatula.

2. Heat the test tube over the burner and observe the change. Note the colour and odour of the gases if evolved as given in Table 2.

### Table 2: Action of Heat

<table>
<thead>
<tr>
<th>Observation</th>
<th>Inference</th>
<th>Chemistry Involved</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonia gas evolves</td>
<td>NH$_4^+$</td>
<td>(NH$_4$)$_2$CO$_3$→NH$_4$+CO$_2$+H$_2$O</td>
</tr>
<tr>
<td>Carbon dioxide evolves</td>
<td>CO$_2^+$</td>
<td>CaCO$_3$→CaO+CO$_2$</td>
</tr>
<tr>
<td>Sulphur dioxide evolves</td>
<td>SO$_4^{2-}$ may be present</td>
<td>2FeSO$_4$→Fe$_2$O$_3$+ SO$_2$+SO$_3$</td>
</tr>
<tr>
<td>Nitrogen dioxide evolves</td>
<td>NO$_3^-$</td>
<td>2Cu(NO$_3$)$_2$→2CuO+4NO$_2$+O$_2$</td>
</tr>
</tbody>
</table>

**Colour of the salt on heating**

1. Some salts change their colour blue to white
2. Green to yellow
3. White (when cold) to yellow (when hot)

**Flame Test**

Prepare a paste of small amount of the salt with one or two drops of conc. HCl on a watch glass. Take the paste on the loop of Nichrome wire or cotton swab, put it in the burner’s flame and observe the colour as given in Table 3.

### Table 3: Flame Test

<table>
<thead>
<tr>
<th>Flame colour observed by naked eye</th>
<th>Inference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deep blue green</td>
<td>Cu$^{2+}$</td>
</tr>
<tr>
<td>Brick red</td>
<td>Ca$^{2+}$</td>
</tr>
<tr>
<td>Apple green</td>
<td>Ba$^{2+}$</td>
</tr>
<tr>
<td>Crimson (deep red)</td>
<td>Sr$^{2+}$</td>
</tr>
</tbody>
</table>

A confirmation is made by other specific tests. Other anions (SO$_4^{2-}$ and PO$_4^{3-}$) are identified by different reagents.

For performing some identification tests for anions, it is essential
to prepare the salt solution. Depending upon the solubility of the given salt, we may prepare its aqueous solution or water extract or sodium carbonate extract for carrying out the tests.

**Water Extract (WE):**

Boil a few mg of the salt with 2 mL distilled water for 2-3 minutes

**Sodium Carbonate Extract (SE):**

If the salt is insoluble in water, its sodium carbonate extract is prepared. On boiling the salt with sodium carbonate solution, double decomposition takes place resulting in the formation of soluble sodium salt of the anion. Mix 5 mg of the salt with 15 mg Na$_2$CO$_3$ in a boiling tube. After transferring this mixture add about 10 mL of distilled water in 25 mL beaker and boil for about 5 min. Filter, the filtrate is the sodium carbonate extract (SE).

**Detection of Cations:**

All cations may be divided into six groups depending upon the difference in solubility of their chlorides, sulphides, hydroxides, carbonates under different conditions of acidity and alkalinity. The reagent used to test the presence of a group is known as a group reagent.

The first step before proceeding to systematic wet analysis of cations is to dissolve the salt in a suitable solvent. The clear solution is known as original solution (OS).

**Instructions:**

1. Note the colour, odour and state of the given salt as given in the Table 1.

<table>
<thead>
<tr>
<th>Physical Properties</th>
<th>Inference</th>
</tr>
</thead>
<tbody>
<tr>
<td>White Colour (colourless)</td>
<td>Pb$^{2+}$, As$^{3+}$, Al$^{3+}$, Zn$^{2+}$, Ca$^{2+}$, Ba$^{2+}$, Sr$^{2+}$, Mg$^{2+}$ or NH$_4^+$ salt may be present.</td>
</tr>
<tr>
<td>Bluish or bluish green colour</td>
<td>Cu$^{2+}$ or Ni$^{2+}$ Salt</td>
</tr>
<tr>
<td>Yellowish brown colour</td>
<td>Fe$^{2+}$ or Ni$^{3+}$ Salt</td>
</tr>
<tr>
<td>Smell of ammonia gas</td>
<td>NH$_3$ Salt</td>
</tr>
<tr>
<td>Smell of hydrogen sulphide gas</td>
<td>S$^2$</td>
</tr>
<tr>
<td>Heavy</td>
<td>Pb$^{2+}$, Ba$^{2+}$ Salt</td>
</tr>
<tr>
<td>Light</td>
<td>Al$^{3+}$, Mg$^{2+}$, Ca$^{2+}$ Salt</td>
</tr>
</tbody>
</table>
Classification of cations in groups: Classification of the cations into groups is based on the values of their solubility products with different anions.

<table>
<thead>
<tr>
<th>Group</th>
<th>Cations</th>
<th>Group Reagent</th>
<th>Precipitated as</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>Pb²⁺, Hg⁺, Ag⁺</td>
<td>Dil HCl</td>
<td>Chlorides - e.g. PbCl₂</td>
</tr>
<tr>
<td>II</td>
<td>Pb²⁺, Cu²⁺, As³⁻</td>
<td>S²⁺ ions in acidic medium</td>
<td>Sulphides - PbS, CuS, As₂S₃</td>
</tr>
<tr>
<td>III</td>
<td>Fe³⁺, Al³⁺</td>
<td>NH₃OH</td>
<td>Fe(OH)₃, Al(OH)₃</td>
</tr>
<tr>
<td>IV</td>
<td>Ni²⁺, Co²⁺, Zn²⁺, Mn²⁺</td>
<td>S²⁺ ions in basic medium</td>
<td>NiS, CoS, ZnS, MnS</td>
</tr>
<tr>
<td>V</td>
<td>Ca²⁺, Ba²⁺, Sr²⁺</td>
<td>(NH₄)₂CO₃</td>
<td>CaCO₃, BaCO₃, SrCO₃</td>
</tr>
<tr>
<td>VI</td>
<td>Mg²⁺, NH₄⁺, NH₃⁺, K⁺</td>
<td>Individual tests</td>
<td></td>
</tr>
</tbody>
</table>

Group VI cations are tested individually with different reagents.

Anion analysis

Anions are classified as follows:

- Dil. H₂SO₄ group
- Conc. H₂SO₄ group
- Independent radicals

\[ \text{CO}_3^{2-}, \text{NO}_2^-, \text{SO}_3^{2-}, \text{SO}_4^{2-}, \text{CH}_3\text{COO} \]

\[ \text{F}^-, \text{Cl}^-, \text{Br}^-, \text{I}^-, \text{NO}_3^- \]

\[ \text{SO}_4^{2-}, \text{PO}_4^{3-} \]

Dil. H₂SO₄ Group:

Take a pinch of salt in a semi micro test tube, add few drops of dil H₂SO₄ and heat. Look for anyone of the following observations:

2. Colourless gas with rotten egg smell - S²⁻ indicated
3. Colourless gas with pungent smell - SO₃²⁻ indicated. Use W-tube for SO₂ test using dil solution of potassium permanganate or dichromate solution.
4. Brown fumes - NO₂⁻ indicated
5. Vinegar smell - CH₃COO⁻ indicated

If anyone of the above is detected, carry out the confirmatory wet test as follows:
1. **Confirmatory test for CO$_3^{2-}$ ion** - Take a pinch of salt in one arm of W-tube and transfer two drops of lime water in the other arm with the help of dropper. Now transfer 2-3 drops of dil HCl with the help of a dropper in the first arm. The gas from this arm bubbles through the arm containing lime water. Lime water turns milky -CO$_3^{2-}$ confirmed.

2. **Confirmatory test for S$_2^-$ ion** - Take few drops of OS in a well in the well plate and add a drop of sodium nitroprusside solution - purple or violet colour confirms S$_2^-$.

3. **Confirmatory test for SO$_3^{2-}$ ion** - Take 4-5 drops of OS and add a few drops of BaCl$_2$ solution – white ppt soluble in dil HCl confirms SO$_3^{2-}$.

4. **Confirmatory test for NO$_2^-$ ion** - (i) Take a few drops of OS in a well in the well plate, add a drop of dil acetic acid followed by a few drops of ferrous sulphate solution — dark brown or black colour confirms NO$_2^-$. (ii) Take 5 mg of solid salt in a micro test-tube and add 3 drops of acetic acid and 5 mg of KI. Warm and shake with 5-6 drops of carbon tetrachloride or chloroform. Violet colour due to iodine in the carbon tetrachloride or chloroform layer confirms presence of nitrite ions. This test can also be done for iodide ions.

5. **Confirmatory test for CH$_3$COO$^-$**
Rub a pinch of the salt with oxalic acid–vinegar smell confirms acetate ion.

**Conc. H$_2$SO$_4$ group**

Take a spatula of salt (10–15 mg) in a micro test tube, add few drops of conc. H$_2$SO$_4$ and heat. Look for anyone of the following observations or Use W-tube for halide ions test taking AgNO$_3$ solution in one arm.

1. Colourless gas with pungent smell, gives dense white fumes when a glass rod dipped in NH$_4$OH is brought near the mouth of test tube - Cl$^-$ indicated.

2. Reddish brown vapours with pungent smell, turn starch paper yellow and do not intensify on adding Cu turning - Br$^-$ indicated.

3. Violet vapours, turn starch solution/paper black -I$^-$ indicated.

4. Brown fumes with pungent smell, intensify on adding a Cu turning -NO$_3^-$ indicated.
If anyone of the above is detected carry out the confirmatory wet test as follows:

1. **Confirmatory test for NO$_3^-$ ion** – Transfer one drop of salt solution in one of the wells of well plate and add one drop of freshly prepared FeSO$_4$ solution. Add 3 drops of conc H$_2$SO$_4$ slowly from the side of the well – a brown ring at the junction of the liquids confirms Cl$^-$.

2. **Confirmatory test for Cl$^-$ ion** – Transfer a small pinch of salt in one of the arms of W-tube and add a pinch of solid K$_2$Cr$_2$O$_7$ followed by 2-3 drops of conc. H$_2$SO$_4$. Transfer 2-3 drops of NaOH solution in the other arm of W-tube. Heat carefully the first arm of the W-tube by holding it by the holder. Now take one drop of this NaOH (yellow in colour) in a well, add a drop of dil acetic acid followed by 2 drops of lead acetate solution – Yellow ppt confirms Cl$^-$.

3. **Confirmatory test for Br$^-$ ion** – Transfer 3-4 drops of OS in one arm of the W-tube and 2 drops of silver nitrate solution in the other arm. Transfer a few drops of dil HNO$_3$ in the first arm with the help of a dropper and warm. Light yellow ppt partially soluble in NH$_4$OH solution confirms Br.

4. **Confirmatory test for I$^-$ ion** – Repeat the above test with salt solution – Yellow ppt insoluble in NH$_4$OH solution confirms I$^-$.

**Independent radicals:**

1. SO$_4^{2-}$ Boil a pinch of salt with small amount of dil HCl (filter if needed to remove any solid present) and add BaCl$_2$ solution – White ppt insoluble in conc HCl confirms SO$_4^{2-}$.

2. PO$_4^{3-}$ Boil a pinch of salt with few drops of conc HNO$_3$ and add ammonium molybdate solution in slight excess – A yellow ppt confirms PO$_4^{3-}$.

**Scheme of classification of cations:**

**Original solution (OS)**: Take a microspatula full of salt in a micro test tube and add 2 mL water. Shake to dissolve the salt. If not soluble, add 2-3 drops of dil. HCl. Warm if necessary.
### GROUP I

| ppt-Gp I present Pb²⁺ Present Test for Pb²⁺ in hot solution Proceed to analyse and confirm Gp I radicals | No ppt—proceed for Group II-Transfer 2 drops of OS solution to a well flask/microtest tube and add 2 drops of Na₂S solution or H₂S in ammonia solution after adjusting the pH to about 2 by adding dil HCl. No ppt seen in the well, proceed for Gp III-Transfer 3–4 drops of OS in microtest tube and add a pinch of solid NH₄Cl, shake dissolve, then add NH₄OH solution, till the solution becomes basic (smell of ammonia). Brown or white ppt-Gp III present brown [Fe⁺⁺] white gelatinous (Al⁺⁺) Proceed to analyse and confirm Gp III radicals | No ppt—proceed for Gp IV—transfer 2 drops of OS to a well plate or test tube and a few drops of Na₂S solution or H₂S in ammonia solution. Black, pink or white ppt-Gp IV present Black ppt-Ni⁺⁺, Co⁺⁺ Proceed to analyse and confirm Gp IV radicals | No ppt—for Gp V—Transfer 5 drops OS in micro test tube and add 3 drops (NH₄)₂CO₃ solution. White ppt-Gp V Present Ca⁺⁺, Ba⁺⁺, Sr⁺⁺ Proceed to analyse and confirm Gp VI radicals | No ppt-Gp VI present Proceed to analyse and confirm Gp VI radicals |

Separate the white ppt and wash with water. Boil the ppt with 1/2 mL water and transfer 2 drops each of the resulting clear solution into two wells in the well plate.

To one well, add one drop of potassium iodide solution-
Yellow ppt confirms Pb²⁺

To the other well, add one drop of potassium chromate solution—
Yellow ppt confirms Pb²⁺
### GROUP II

<table>
<thead>
<tr>
<th>Yellow ppt As³⁺ present</th>
<th>Black ppt-Pb²⁺ or Cu²⁺ present</th>
</tr>
</thead>
<tbody>
<tr>
<td>Confirmation</td>
<td></td>
</tr>
<tr>
<td>Dissolve the ppt in min quantity of conc HNO₃. Add ammonium Molybdate solution and heat.</td>
<td></td>
</tr>
<tr>
<td>Yellow ppt confirms As³⁺</td>
<td></td>
</tr>
</tbody>
</table>

- **Confirmation**
  - Transfer 1 drop of OS in well plate and add 1 drop of H₂SO₄ and 1 drop of alcohol.
  - White ppt- Pb²⁺ indicated.
  - Do other tests for Pb²⁺ as given in Group 1 table.
  - To the other part of solution, add NH₄OH in excess.
  - Blue coloured solution-Cu²⁺ indicated.
  - **Confirmation**
    - Transfer 2 drops of blue solution to two wells in the well plate:
      1. To one well, add a drop of acetic acid followed by a drop of potassium ferrocyanide solution.
      - **Chocolate brown ppt-Cu²⁺ confirmed.**
      2. To the other well, add a drop of acetic acid followed by a drop of potassium iodide solution.
      - **Dirty yellow ppt-Cu²⁺ confirmed.**
    - Add 3 drops of CCl₄/CHCl₃ and shake, violet colour in organic layer-Cu²⁺ confirmed.

### GROUP III

Transfer 4 drops of OS in a micro test-tube and add a pinch of ammonium chloride followed by 2 drops of ammonium hydroxide.

**Confirmation**

1. Brown ppt. Transfer 2 drops of OS to a well in the well plate and add a drop of potassium ferrocyanide solution. Prussian blue colour-Fe³⁺ confirmed.
2. Transfer one drop of OS in well plate and add 1 drop of KSCN/NH₄SCN-Blood red colour-Fe³⁺ confirmed.
GROUP IV

<table>
<thead>
<tr>
<th>Co²⁺ indicated</th>
<th>Ni²⁺ indicated</th>
<th>Zn²⁺ indicated</th>
<th>Mn²⁺ indicated</th>
</tr>
</thead>
<tbody>
<tr>
<td>Confirmation—</td>
<td>Confirmation—</td>
<td>Confirmation—</td>
<td>Confirmation—</td>
</tr>
<tr>
<td>Transfer 2-3 drops of OS and a crystal of ammonium thiocyanate-Blue colour in the either layer confirms Co²⁺. Transfer 2 drops of OS in well plate and add 2 drops of NH₄SCN and amyl alcohol-blue colour in amyl alcohol layer.</td>
<td>Transfer 2 drops of OS in a well of well plate, add a drop of dimethylglyoxime solution and NH₄OH Pink ppt confirms Ni²⁺.</td>
<td>Transfer 3 drops of OS to a well and add NaOH drop wise-white ppt which on addition of Br₂ water turns black or changes without Br₂, the ppt drops of dil HCl and colour on standing for some time. Mn²⁺ confirmed.</td>
<td>Transfer 2 drops of OS to a well and add NaOH drop wise-white ppt which on addition of Br₂ water turns black or changes without Br₂, the ppt drops of dil HCl and colour on standing for some time. Mn²⁺ confirmed.</td>
</tr>
</tbody>
</table>

GROUP V

White ppt indicates Ba²⁺ or Sr²⁺ or Ca²⁺. Separate the ppt and wash with water. Dissolve in hot dil acetic acid and divide the solution into three parts. Confirmation to be carried out in the sequence given below.

<table>
<thead>
<tr>
<th>Ba²⁺</th>
<th>Sr²⁺</th>
<th>Ca²⁺</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Transfer 2 drops of solution to a well and add 2 drops of potassium chromate solution—Yellow ppt confirms Ba²⁺. 2. Perform flame test-dip a cotton swab (ear bud) in water and touch it to the salt. Heat in the reducing flame—Apple green flame confirms Ba²⁺.</td>
<td>1. To a part of the solution add 2 drops of ammonium sulphate solution in a micro test tube and warm-White ppt confirms Sr²⁺. 2. Perform flame test-dip a cotton swab (ear bud) in water and touch it to the salt. Heat in the reducing flame—Crimson red flame confirms Sr²⁺.</td>
<td>1. To a part of solution, add 2 drops of ammonium oxalate solution in a test tube. Add a few drops of NH₄OH and scratch the side of test tube-White ppt confirms Ca²⁺. 2. Perform flame test-dip a cotton swab (ear bud) in water and touch it to the salt. Heat in the reducing flame—Brick red flame confirms Ca²⁺.</td>
</tr>
</tbody>
</table>
Comments

The following points should be kept in mind while using micro kit for qualitative analysis:

1. Solution (original) (OS) should be prepared in micro test tubes.
2. Whenever heating is required, prefer test tubes over well plates.
3. For confirmatory tests, prefer well plates.
4. For all gas confirmation, use W- Tube e.g. CO$_3^{2-}$, NO$_2^-$, etc.

<table>
<thead>
<tr>
<th>Group VI</th>
<th>If no ppt is formed in Gp V test, Mg$^{2+}$ or NH$_4^+$ may be present</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mg$^{2+}$</td>
</tr>
<tr>
<td>Transfer 6-7 drops of OS in a micro test-tube, add a pinch of solid NH$_4$Cl, shake and add NH$_4$OH in slight excess. Add a few drops of ammonium phosphate solution (or any other soluble or ammonium hydrogen phosphate) and rub the side of the test tube with a glass rod.</td>
<td>1. Take one micro spatula full of salt in a micro test-tube and add a few drops of strong solution of NaOH, and heat – Gas smelling of NH$_3$ indicates NH$_4^+$ ion.</td>
</tr>
<tr>
<td>A white ppt confirms Mg$^{2+}$</td>
<td>2. Suck the gas evolved in the above test using a dropper. Bubble the gas into drops of Nessler's reagent taken in a well of well plate – Brown ppt confirms NH$_4^+$</td>
</tr>
</tbody>
</table>

**Table:**

<table>
<thead>
<tr>
<th>Mg$^{2+}$</th>
<th>NH$_4^+$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Transfer 6-7 drops of OS in a micro test-tube, add a pinch of solid NH$_4$Cl, shake and add NH$_4$OH in slight excess. Add a few drops of ammonium phosphate solution (or any other soluble or ammonium hydrogen phosphate) and rub the side of the test tube with a glass rod.</td>
<td>1. Take one micro spatula full of salt in a micro test-tube and add a few drops of strong solution of NaOH, and heat – Gas smelling of NH$_3$ indicates NH$_4^+$ ion.</td>
</tr>
<tr>
<td>A white ppt confirms Mg$^{2+}$</td>
<td>2. Suck the gas evolved in the above test using a dropper. Bubble the gas into drops of Nessler’s reagent taken in a well of well plate – Brown ppt confirms NH$_4^+$</td>
</tr>
</tbody>
</table>
3.2 Class XII

Surface Chemistry

Experiment 1: To prepare lyophobic and lyophilic sols for the following-

(1) Fe(OH)$_2$  (2)As$_2$S$_3$  (3)Al(OH)$_3$  (4) Starch

Apparatus required:
Beakers (10 mL and 50 mL), measuring cylinder, kerosene burner with tripod, glass rod, Pasteur pipette/dropper, micro test tube with stand and china dish.

Chemicals required:
FeCl$_3$ (anhydrous), AlCl$_3$ (anhydrous), As$_2$O$_3$, Starch, Gum, Egg albumin, H$_2$S (aq), A$_2$(SO$_4$)$_3$, NaCl and parchment paper.

Procedure:
A1. Preparation of lyophilic sol

1. Egg albumin sol-
   i. Prepare 10 mL 15 % (w/v) solution of NaCl in a 50 mL beaker. Break one egg in a china dish and suck 10 mL of the albumin with the help of Pasteur pipette and pour it in sodium chloride solution. Stir it well to ensure that the sol has been well prepared. You may compare it with egg albumin.

2. Starch/ gum sol.
   i. Measure 20 mL distilled water with the help of measuring cylinder and transfer it to 50 mL beaker and boil it.
   ii. Make a paste of starch or gum in water and transfer a small portion of this paste in the boiling water with constant stirring.

II. Preparation of Lyophobic sol.

1. Ferric hydroxide / Aluminium hydroxide -
   i. Take 5 mL distilled water in a 10 mL beaker and boil it.
   ii. Add few drops of 3 % ferric chloride/ aluminium chloride solution to the boiling water and stir it well. Fe(OH)$_3$ solution is formed.
2. Arsenious Sulphide Sol

1. Transfer 5 mL distilled water in 10 mL beaker and boil.
2. Add 0.2 g arsenious oxide to water (procured in step 1) and boil the contents.
3. Cool and filter the solution.
4. Pass H\textsubscript{2}S gas though the filtered solution till the solution gives smell of H\textsubscript{2}S.
5. Expel H\textsubscript{2}S gas from the sol by slow heating and filter it.
6. Label it as arsenious sulphide sol.

B. Purification of the Sol (Dialysis)

1. Take a square sheet (30 cm × 30 cm) of parchment paper.
2. Soak the sheet in water and give it conical shape.
3. Pour the desired colloidal of starch, gum, egg albumin etc. in the cone of parchment/cellophane paper. (Check if the colloidal solution contains some ionic substance e.g Na\textsuperscript+ and Cl\textsuperscript− as an impurity in it)

(1) After about half an hour, test the presence of ions present as the impurity in the water.

(2) Change the water after every half an hour till it is free from impurity of ions. Na\textsuperscript+ and Cl\textsuperscript−

(3) Write the time taken for the purification of each colloidal solution on the basis of your observation.
## Chemical Kinetics

**Experiment 2: To study the effect of temperature on the rate of the reaction between sodium thiosulphate and hydrochloric acid.**

**Apparatus required:**
Stop watch, thermometer, volumetric flask-2 (25 mL), beakers (10 mL), kerosene burner, china dish, tripod.

**Chemicals Required:**
0.2 M Na$_2$S$_2$O$_3$ and 1 M HCl

**Procedure:**
1. Prepare 0.2 M Na$_2$S$_2$O$_3$ in a 25mL flask and 1.0 M HCl in another 25 mL flask.
2. Take equal amounts of 0.2 M Na$_2$S$_2$O$_3$ and 1.0 M HCl in a beaker (10 mL) (3.0 mL each)
3. Record the time taken for appearance of turbidity at room temperature.
4. Repeat the experiment at 30°, 40° and 50°C and record the time taken for completion of reaction.

**Observations:**

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Temperature of the reaction mixture (°C)</th>
<th>Volume of Na$_2$S$_2$O$_3$ (mL)</th>
<th>Volume of HCl (mL)</th>
<th>Time taken for completion of reaction in sec</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>21°C</td>
<td>3.0</td>
<td>3.0</td>
<td>—</td>
</tr>
<tr>
<td>2.</td>
<td>30</td>
<td>3.0</td>
<td>3.0</td>
<td>—</td>
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<tr>
<td>3.</td>
<td>40</td>
<td>3.0</td>
<td>3.0</td>
<td>—</td>
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<tr>
<td>4.</td>
<td>50</td>
<td>3.0</td>
<td>3.0</td>
<td>—</td>
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</table>

**Conclusion**

**Result:**

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Experiment 3: To study the effect of variation of concentration on the rate of reaction between sodium thiosulphate and hydrochloric acid.

Apparatus required:
Micro test tubes -7, test tube stand, volumetric flask (25mL)-2, Pasteur pipette-1, micro beaker -7.

Chemicals required:
0.2 M Na$_2$S$_2$O$_3$ and 1 M HCl

Procedure:
1. Prepare 0.2 M Na$_2$S$_2$O$_3$ solution and 1 M HCl solution in two 25 mL flasks.
2. Take 7 micro test tubes marked as 1,2,3,4,5,6,7.
3. In microtest tube 1, transfer seven drops of 0.2 M Na$_2$S$_2$O$_3$, in test tube 2 transfer 6 drops, in test tube 3, transfer 5 drops and so on.
4. Make up the volume of each test tube equal to the test tube 1 by transferring 1 drop of water to test tube 2, 2 drops of water test tube 3 and so on.
5. Place all of these test tubes in the test-tube stand over a white paper. The white paper should have marks of 'X' under each test tube.
6. Transfer 2 drops of IM HCl in each test tube.
7. Record the time for each test tube when the visibility of the X mark disappeared due to the formation of colloidal sulphur.

Note: HCl should be transferred in each test tube in one pour and not drop wise. You can use micro beakers instead of test tubes.
## Observations

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Drops of Na₂S₂O₃ (mL)</th>
<th>Drops of H₂O (mL)</th>
<th>Drops of IM HCl (mL)</th>
<th>Time taken for the completion of reaction (s)</th>
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<tbody>
<tr>
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</table>

**Result:**

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Experiment 4: To study the effect of variation of concentration on the rate of reaction of iodide ions with hydrogen peroxide at room temperature.

Apparatus required:
Micro test tubes with stand-8 or well plate or microbeakers, volumetric flasks (25 mL) -4, measuring cylinder (10 mL), beaker( 50 mL) and Pasteur pipette.

Chemicals required:
2.5 M H₂SO₄, 0.1 M KI, 0.04 M Na₂S₂O₃ and 3% H₂O₂

Procedure:
1. Transfer 5 mL of hydrogen peroxide in 50 mL beaker. Transfer 5 mL of 2.5 M H₂SO₄, 2 drops of freshly prepared starch solution and 20 mL distilled water to it. Stir the solution well.
2. Take a well plate or micro test tubes with stand or micro beakers.
3. In well 1, transfer 1 drop of 0.04 M sodium thiosulphate solution, 1 drop of 0.1 M potassium iodide solution and 8 drops of distilled water.
4. In well 2, transfer 1 drop of 0.04 M sodium thiosulphate solution, 2 drops of 0.1 M potassium iodide solution and 7 drops of distilled water.
5. In well 3, transfer 1 drop of 0.04 M sodium thiosulphate solution, 3 drops of 0.1 M potassium iodide solution and 6 drops of distilled water.
6. Take a 10 mL beaker and mark it as 4. Transfer 5 drops of solution from the 50 mL beaker (in step 1). Now add 5 drops of solution from well stir, the solution well and record the time required for appearance of blue colour.
7. In exactly the same way, repeat the experiment with the solutions of wells 2 and 3 by using 5 drops of the solution of each of these wells.
8. Repeat the experiment two times and calculate the average time for the appearance of blue colour.

Result:
Experiment 5: To study the rate of reaction between potassium iodate (KIO₃) and sodium sulphite (Na₂SO₃) using starch solution as Indicator.

Apparatus required:
Pasteur pipette, micro beaker(10 mL)-5, beakers 50 mL-2, kerosene burner and tripod.

Chemicals required:
1% Starch solution, 0.2% Na₂SO₃ solution, H₂O, 0.2% KIO₃ and Conc. H₂SO₄

Procedure:
1. Preparation of the standard solution -
   (a) For 0.2% KIO₃ solution - take 0.1 g of KIO₃ in 10 mL water in 0.62 mL of 0.5 M H₂SO₄ and make 50 mL solution by adding water.
   (b) For 0.2% Na₂SO₃ solution - take 0.08 g of Na₂SO₃ in 25 mL water.
   (c) For 1% Starch - take 0.25 g starch (soluble) in 25 mL boiled water.
2. Take 5 beakers(10 mL) marked 1,2,3,4 and 5.
3. (a) In beaker 1, transfer 1 mL of KIO₃ solution, 1 mL of 1% starch solution and 3 mL of water to make 5 mL solution. Mix it well.
   (b) In beaker 2, transfer 2 mL of KIO₃ solution, 1 mL of 1% starch solution and 2 mL of H₂O to make 5 mL solution. Mix it well.
   (c) In beaker 3, transfer 3 mL of KIO₃, 1 mL of 1% starch solution and 1 mL of H₂O to make 5 mL solution. Mix it well.
   (d) In beaker 4, transfer 4 mL of KIO₃ solution, 5 mL of 0.2% starch solution to make 5 mL solution. Mix it well.
   (e) In beaker 5, transfer 4.5 mL of KIO₃ solution, 0.5 mL of starch solution. Mix it well.
4. Transfer 5 mL 0.2% Na₂SO₃ solution in each beaker and record the time required for appearance of blue colour in each case.
<table>
<thead>
<tr>
<th>S.No.</th>
<th>KIO₃ (mL)</th>
<th>Starch (mL)</th>
<th>Water (mL)</th>
<th>Na₂S₂O₃ (mL)</th>
<th>Time taken (s)</th>
</tr>
</thead>
<tbody>
<tr>
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</table>

**Result:**

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Thermochemistry

Experiment 6: To determine the enthalpy of solution

Apparatus required
Calorimeter (50 mL beaker inserted in LDPE foam case), thermometer (0.1°C), stirrer, beaker (50 mL) -2, tripod and kerosene burner.

Chemical required
CuSO\textsubscript{4}.5 H\textsubscript{2}O

Procedure

Determination of Water Equivalent of Calorimeter-
1. Take a beaker of 50 mL capacity. Weigh it. Let its mass be \( m_1 \) g. Note that 50 mL beaker is inserted in LDPE foam case to make calorimeter.
2. Take 10 mL water in the beaker and again weigh it. Let the mass this time be \( m_2 \) g.
3. Record the temperature of water. Let this temperature be \( T_1 \) °C.
4. Place it in the LDPE foam case. Insert carefully the stirrer in the lid and then thermometer through circular part of the stirrer. Adjust the height of the thermometer and stirrer to fit in the case.
5. In another beaker, take 10 mL of hot water (50°−60° C) and weigh it. Let the mass of hot water be \( m_3 \) g and temperature be \( T_2 \) °C.
6. Note the exact temperature of hot water.
7. Transfer the hot water from beaker to the beaker in the calorimeter. Place the lid and stir the mixed water and note its temperature. Let this temperature be \( T_3 \) °C.
8. Calculate water equivalent of the beaker by using the expression
   \[
   (m_2-m_1)+W(T_3-T_1)=m_3(T_2-T_3).
   \]
   (Remember the three temperatures are in the order \( T_2>T_3>T_1 \).
   Thus the above equation gives \( W \), the water equivalent of the calorimeter.

Determination of enthalpy of solution:
1. Transfer 20 mL distilled water in the 50 mL beaker placed in the cavity of LDPE foam case.
2. Record the temperature of water. Let this be $T_1 \degree C$.

3. Transfer powdered and weighed amount of copper sulphate. Stir the solution till the entire copper sulphate dissolves. Keep the thermometer and stirrer intact on the mouth of the foam case.

4. Note down the maximum temperature attained by the solution after the addition of copper sulphate. Let this be $T_2 \degree C$.

Observations:

- Mass of the cold water = 20 g
- Mass of the copper sulphate = mg
- Temperature of the cold water = $T_1 \degree C$
- Temperature of the mixture = $T_2 \degree C$

Calculations:

Now calculate the enthalpy of solution

Total mass of the solution = Mass of water + Mass of copper sulphate = $(20 + m)$ g

Rise in temperature = $(T_2 - T_1)$ $\degree C$

Heat gained by the calorimeter = $W(T_2 - T_1)$ $\degree C$

Heat gained by the solution = $[(20 + m)(T_2 - T_1) + W(T_2 - T_1)] \times 4.18J$

Heat liberated on account of dissolution of 1 g copper sulphate =

$$\frac{[W(T_2 - T_1)(20 + m)(T_2 - T_1)]}{m} \times 4.18$$

$$\text{sol H (Copper sulphate)} = \frac{[W(T_2 - T_1)(20 + m)(T_2 - T_1)]}{m} \times M \times 4.8 \text{J mol}^{-1}$$

Where $M$ is the Molar mass of Copper sulphate

Result:
**Experiment 7: To determine the enthalpy of neutralisation of reaction between 1M HCl and 1M NaOH.**

**Apparatus required:**
Calorimeter, beaker(50 mL)-2, thermometer (0.1°C), stirrer, kerosene burner and tripod

**Chemicals required:**
IM HCl and IM NaOH

**Procedure:**

**Determination of water equivalent of the calorimeter**
This may be determined by following the procedure given in Experiment 6.

**Determination of enthalpy of neutralisation**

1. Take 10 mL of 1M HCl solution in the calorimeter and also 10 mL 1M NaOH in another beaker of 50 mL capacity.
2. Note the temperature of both the solutions, which are likely to be the same. Let it be $T_1$°C.
3. Transfer 10 mL IM NaOH solution to the calorimeter. Put the lid in place.
4. Stir the solution rapidly and note the final temperature of the mixture. Let it be $T_2$°C.

**Calculation:**

(i) Calculation of the water equivalent ($W$) of the calorimeter-as done in Experiment 6.

(ii) Calculation of enthalpy of neutralization

Temperature of 10 mL HCl in 50 mL beaker in the calorimeter = $T_1$°C

Final temperature of the solution = $T_2$°C

Rise in temperature = $(T_2 - T_1)$°C
Heat produced during neutralisation = \((10+10+W)(T_2-T_1) \times 4.18J\)

Determine the heat produced when 1000 mL 1M HCl is allowed to neutralize 1000 mL 1M NaOH. This quantity would be 100 times the quantity obtained above. Express the quantity of heat evolved in kJ/mol.

**Result:**

\[ \text{Result} \]

\[ \text{Result} \]

\[ \text{Result} \]

\[ \text{Result} \]

\[ \text{Result} \]
Electrochemistry

Experiment 8: To study the variation of cell potential of the cell
Zn / Zn⁺⁺ || Cu⁺⁺ / Cu with the change in the concentration of
electrolytes at the room temperature.

Apparatus required:
Beakers(50 mL) -2, measuring flasks(25 mL)-2, Y-tube/filter paper strip / wick, rubber bulb, Cu and Zn strips and emery paper.

Chemical required
1M ZnSO₄ and 1M CuSO₄

Procedure
1. Prepare 1M ZnSO₄ and 1M CuSO₄ in the measuring flasks.
2. Transfer the solutions to separate 50 mL beakers.
3. Prepare about 20 mL saturated solution of potassium chloride. Fill this solution in the Y shaped tube with the help of rubber bulb. The filled solution in this tube should be carried carefully to dip the arms in the two beakers as shown. You can take the filter paper strip dipped in KCl instead of Y shaped tube.
4. Clean the Cu and Zn strips with emery paper. Place Zn strip in ZnSO₄ solution and Cu strip in CuSO₄ solution.
5. Measure the cell emf with the help of multimeter setting the knob of the multimeter at 20 V range. (Note that +ve lead (red) should be inserted in the + ve and - ve lead (black) in common hole of the multimeter.
6. Repeat the procedure with change in concentration of copper sulphate or zinc sulphate solution.
Observations:

Table 1 [Zn⁺⁺] = IM

<table>
<thead>
<tr>
<th>S.No.</th>
<th><a href="M">Cu⁺⁺</a></th>
<th>log[Cu⁺⁺]</th>
<th>Obs. E_{cell}/V</th>
<th>Calculated E_{cell}</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>1.0</td>
<td>0</td>
<td>0.98</td>
<td>0.98</td>
</tr>
<tr>
<td>2.</td>
<td>0.5</td>
<td>-0.301</td>
<td>0.96</td>
<td>0.97115</td>
</tr>
<tr>
<td>3.</td>
<td>0.33</td>
<td>-0.5</td>
<td>0.95</td>
<td>0.96525</td>
</tr>
<tr>
<td>4.</td>
<td>0.2</td>
<td>-0.699</td>
<td>0.95</td>
<td>0.96935</td>
</tr>
<tr>
<td>5.</td>
<td>0.1</td>
<td>-1</td>
<td>0.94</td>
<td>0.9505</td>
</tr>
</tbody>
</table>

Table 2 [Cu⁺⁺] = IM

<table>
<thead>
<tr>
<th>S.No.</th>
<th>[Zn⁺⁺]</th>
<th>log[Zn⁺⁺]</th>
<th>Obs. E_{cell}</th>
<th>Calculated E_{cell}</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>1.0</td>
<td>0</td>
<td>0.98</td>
<td>0.98</td>
</tr>
<tr>
<td>2.</td>
<td>0.5</td>
<td>-0.31</td>
<td>1.04</td>
<td>0.98555</td>
</tr>
<tr>
<td>3.</td>
<td>0.25</td>
<td>-0.6</td>
<td>1.06</td>
<td>0.99775</td>
</tr>
</tbody>
</table>

Table 3 [Cu⁺⁺] = IM

<table>
<thead>
<tr>
<th>S.No.</th>
<th><a href="M">Cu⁺⁺</a></th>
<th>log[Cu⁺⁺]</th>
<th>Obs. E_{cell}</th>
<th>Calculated E_{cell}</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>1.0</td>
<td>0</td>
<td>1.0V</td>
<td>1.0V</td>
</tr>
<tr>
<td>2.</td>
<td>0.1</td>
<td>-1</td>
<td>0.94</td>
<td>0.9705</td>
</tr>
<tr>
<td>3.</td>
<td>0.01</td>
<td>-2</td>
<td>0.92</td>
<td>0.9410</td>
</tr>
<tr>
<td>4.</td>
<td>0.001</td>
<td>-3</td>
<td>0.90</td>
<td>0.9115</td>
</tr>
<tr>
<td>5.</td>
<td>0.0001</td>
<td>-4</td>
<td>0.89</td>
<td>0.8810</td>
</tr>
</tbody>
</table>

Result:
Chromatography

Experiment 9: To study the separation of pigments from extracts of leaves and flowers by paper chromatography and determination of $R_f$ values.

Apparatus required:

Chromatography jar with cork and hook, strip of filter paper-6 mm x 8 cm, Pasteur pipette, leaf/flower/ink and black permanent marking pen

Chemicals required:

Ethanol

Procedure

Part-1

1. Use a black permanent marking pen to make a small ink dot about 5 cm away from one edge of the 8 cm long strip of filter paper or put spots of extracts of leaves, flower etc. Mount it on the hook of the cork to be placed on the mouth of the chromatography jar.

2. Transfer 5 to 6 mL of ethanol into the jar.

3. Carefully insert the filter paper into the jar so that the lower end below the masks dips in ethano.

4. Wait for about 10 minutes.

Part-2

1. Repeat steps 1 to 3 as in part 1, using a new strip of filter paper and water as the solvent.

2. Wait for about 10 minutes. Calculate $R_f$ values.

Result:

<table>
<thead>
<tr>
<th>Fig.9.1: Arrangement for vertical chromatography</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cook</td>
</tr>
<tr>
<td>hook</td>
</tr>
<tr>
<td>chromatography jar</td>
</tr>
<tr>
<td>filter paper strip</td>
</tr>
<tr>
<td>Black inkdot</td>
</tr>
<tr>
<td>Ethanol</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Fig.9.1: Arrangement for vertical chromatography</th>
<th>Question: What is the purpose of using a black permanent marking pen?</th>
</tr>
</thead>
<tbody>
<tr>
<td>Use a black permanent marking pen to make a small ink dot about 5 cm away from one edge of the 8 cm long strip of filter paper or put spots of extracts of leaves, flower etc.</td>
<td>Make ink dots or spots for the chromatography process.</td>
</tr>
</tbody>
</table>

Observations

<table>
<thead>
<tr>
<th>Question: What is the purpose of using ethanol as a solvent?</th>
</tr>
</thead>
<tbody>
<tr>
<td>To separate pigments from extracts of leaves and flowers.</td>
</tr>
</tbody>
</table>
Experiment 10: To separate the constituents present in an inorganic mixture containing two cations only (Cu\textsuperscript{2+}, Cd\textsuperscript{2+}) using the technique of chromatography.

**Apparatus required:**

Petridish-2, cotton, circular Whatman paper

**Chemicals required:**

Solution of mixture of above two cations (Cu\textsuperscript{2+}, Cd\textsuperscript{2+}), Sodium sulphide solution and ethanol, 6 M HCl as solvent.

**Procedure:**

1. Put a spot of the mixture on the marked centre of the circular paper.
2. Make a pointed wick of cotton.
3. Insert the tip of the wick in the centre of the paper with the help of a pin where the spot is put.
4. Keep the base of the wick in the solvent taken in the petridish and let it stand for a while. Cover the paper with another petridish.
5. Take out the paper (Chromatogram), mark the solvent phase and dry it. Spray sodium sulphide solution on it.
6. Calculate $R_f$ value of both components (cations)
<table>
<thead>
<tr>
<th>S.No.</th>
<th>Colour of the spots of ions</th>
<th>Distance travelled by the components of the spots from the reference line</th>
<th>Distance travelled by the solvent from the reference line</th>
<th>$R_f$ value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Result:**
Preparation of Inorganic Compounds

Experiment 11: To prepare inorganic compounds.

1. Preparation of a double salt-potash alum

Apparatus required:
Beaker (50 mL), glass rod, filtration apparatus, tripod and kerosene burner

Chemicals required:
Potassium sulphate, aluminium sulphate, dil. sulphuric acid

Procedure:

1. Dissolve 1.7 gm of potassium sulphate in 10 mL distilled water with 3–5 drops of dil. sulphuric acid.
2. Dissolve 2.5 g of aluminium sulphate in 10 mL distilled water in a separate beaker.

3. Mix the two solutions and filter them using the filtration apparatus.

4. Concentrate the solution by heating while stirring it thoroughly.

5. Allow the solution to cool to the room temperature slowly. On cooling, crystals of potash alum will separate out.

6. Separate the crystals by filtration, wash with cold water, dry between the folds of filter paper and record the yield.

**2. Preparation of potassium ferric oxalate**

*Apparatus required:*

- Beaker (50 mL)
- China dish
- Filtration unit
- Kerosene burner
- Tripod
- Glass rod

*Chemicals required:*

- Ferrous sulphate
- Ammonium sulphate
- $\text{H}_2\text{SO}_4$
- Oxalic acid
- Potassium hydroxide
- Ferric chloride

*Procedure:*

1. Prepare oxalic acid solution by taking 2.9 g of oxalic acid in 10 mL hot water in a clean 50 mL beaker.

2. To the above solution, add 2.58 g of potassium hydroxide gradually in slots with stirring so that it dissolves completely.

3. Now add 1.25 g of ferric chloride into the reaction mixture with constant stirring till it completely dissolves.

4. Filter the solution and concentrate the green filtrate in a china dish over the burner and cool.

5. Filter the compound formed, wash with cold water and make it dry by pressing between the folds of filter paper.

6. Record the yield.
Preparation of Organic Compounds

Experiment 12: To prepare the following organic compounds—

1. Iodoform
2. Acetanilide
3. Dibenzalacetone
4. p-nitro acetanilide

Apparatus required:
Beakers (50 mL and 10 mL), filtration unit, dropper, kerosene burner, tripod, stirrer, china dish and round bottom flask

Chemicals required:
Acetone, KOH, dioxane, iodine solution, aniline, activated charcoal, KI, sodium acetate, acetic anhydride, HBr and benzaldehyde

Procedure:
1. **Preparation of Iodoform**
   1. Take 10 drops of acetone in a beaker and add 20 drops of 10% KOH solution to it.
   2. Warm the mixture in a water bath.
   3. Transfer 20 drops of iodine solution and stir it properly.
   4. Now add 20 drops of 10% KOH solution dropwise.
   5. Warm the solution for two minutes and cool. Light yellow precipitate of iodoform is formed.
   6. Filter, dry and find the yield.

2. **Preparation of Acetanilide**
   1. Take two drops of aniline and add 1 mL of water in a beaker.
   2. Add three drops of acetic anhydride solution dropwise and stir with a thin glass rod thoroughly.
   3. Allow the mixture to stand at room temperature. White crystalline acetanilide is obtained.

3. **Preparation of Dibenzalacetone**
   1. Prepare a mixture of 1.0 mL of benzaldehyde and 0.5 mL of acetone in a micro beaker.
2. Take a mixture of 10.0 mL of 5 M NaOH and 8.0 mL of ethanol in a round bottom flask which can be stoppered with a cork stopper.

3. Place the flask in china dish having some cold water (temperature maintained around 20°C).

4. Add the aldehyde -ketone mixture with the help of dropper in in the interval of five minutes.

5. Stir the reaction mixture for about 30 minutes in the water bath maintained at below 25°C.

6. Filter the product under suction using filtration unit. Dry and note its yield.

4. **Preparation of p-nitroacetanilide**

1. Add 1.0 g acetanilide to 1 mL of glacial acetic acid in a micro beaker and add 2.0 mL of Conc. H₂SO₄ with stirring.

2. Cool the solution to 0-2°C.

3. Add the mixture of 0.44 mL of conc. HNO₃ and 0.28 mL of conc.H₂SO₄ with the constant stirring and maintaining the temperature.

4. Allow it to stand at room temperature for an hour.

5. Pour the mixture in 20 mL cold water and filter the solid, wash with water.

6. Crystalline p-nitroacetanilide is obtained.
Functional Group tests in Organic compounds

Experiment 13: To test functional groups present in an organic compound

To identify the following functional group present in an organic compound-

1. Unsaturation
2. Alcoholic
3. Phenolic
4. Aldehydic
5. Ketonic
6. Carboxylec
7. Amino group (primary)

Apparatus required:
Well plate, micro test tubes, burner and W-tube

Chemicals required:
(a) Solid reagents- Calcium carbide
(b) Liquid reagents-
Alkaline solution of KMnO₄, Fehling solution A, Fehling solution B, Tollens reagent, Schiffs reagent, aniline, dil. HCl, aq. NaNO₂, β-naphthol solution.

Procedure:

(A) Test of unsaturation

Baeyer’s Test-
1. Take a spatula of CaC₂ in one side of W-tube and in the other side a very dilute alkaline KMnO₄ solution.
2. Add a few drops of distilled water to the side containing CaC₂ and close the mouth by your thumb. The produced gas acetylene goes into other side and reacts with KMnO₄ solution.
3. Dil. KMnO₄ solution gets decolourised within few seconds.
(B) Test for Aldehyde-
(I). Schiff’s reagent Test -
(1) Take 3-4 drops of the liquid or 50 mg of solid organic compound in a micro test tube or well plate.
(2) Add 2-3 drops of the Schiff’s reagent.
(3) Appearance of pink colour indicates the presence of an aldehyde.

(II). Fehling’s solution test-
(1) Take 1 mL of both Fehling solution A and 1 mL of Fehling solution B in micro test tube.
(2) Add 2-3 drops of organic compound
(3) Heat the content for about 2 minutes on a water bath.
(4) Formation of a brick red ppt. of cuprous oxide indicates the presence of aldehyde.
(5) This test is not given by the aromatic aldehydes.

(III). Tollen’s reagent test-
(1) Take 1 mL of freshly prepared silver nitrate (2 %) in a test tube. Add 1-2 drops of sodium hydroxide solution to it and shake. A dark brown ppt. of silver oxide appears. Dissolve the ppt. by adding ammonium hydroxide solution dropwise.
(2) Now, add an aqueous or alcoholic solution of about 50 mg of organic compound.

(3) Heat the reaction mixture on a water bath for 5 minutes.

(4) Formation of a deposit of silver, on the inner side of the test tube indicates the presence of aldehyde.

(C) **Test for carboxylic group**

*Sodium bicarbonate test*

(1) Take 2 mL of approx. 5% of aq. solution of sodium bicarbonate in a test tube.

(2) Add few drops of organic compound.

(3) The evolution of brisk effervescence of CO₂ indicates the presence of carboxylic compound.

(D) **Test for amino groups (primary aromatic amine)**

*Azo dye test*

(1) Take one drop of aniline in small test tube and add NaNO₂ solution and dil. HCl.

(2) Cool the contents of the test tube in ice while maintaining the temperature of the mixture to about 0–5°C.

(3) In another test tube, take β-napthol solution and dissolve it in the dil. NaOH solution.

(4) Add diazonium chloride solution slowly with shaking.

(5) Formation of an orange dye confirms the presence of an aromatic primary amino compound.
Experiment 14: To characterise carbohydrates, fats and proteins in pure form and detect their presence in given food stuffs.

Apparatus required
Well plates, microtest tubes-10, test tube stand, Pasteur pipette, dropper, micro spatula, kerosene burner, tripod and china dish.

Chemicals required:
Molisch’s reagent, conc. H₂SO₄, conc. HNO₃, Benedict’s solution, Fehling solution A, Fehling solution B, Tollens reagent, iodine solution, starch solution, KHSO₄, CuSO₄ solution, potassium hydrogen sulphate.

Food stuffs required:
Gram, ground nut, oil, milk, and grape juice

Procedure:
I. Test for carbohydrates
Dissolve a spatula of glucose in 2 mL of water and transfer 2 drops of the solution into each of four micro test tubes 1, 2, 3, 4.

(1) Molisch test:
(1) In 1, dispense 2-3 drops of naphthol solution and then add 2-3 drops of conc. H₂SO₄ from the sides.

(2) Formation of purple ring at the interface of two layers confirms the presence of carbohydrates.

Test for reducing sugars
1. Fehling solution test
(1) Take 1 mL each of Fehling solution A and 1 mL of Fehling solution B in a test tube.
(2) Add 2-3 drops of organic compound
(3) Heat the contents for about 2 minutes on a water bath.
(4) Formation of a brick red ppt. of cuprous oxide indicates the presence of carbohydrates with reducing property such as glucose and fructose.
2. **Benedict's test**

(1) Add benedicts reagent to test tube 3 and heat.

(2) Formation of red ppt due to account of the formation of copper oxide indicates the presence of carbohydrates with reducing property e.g. glucose and fructose.

3. **Tollen's reagent test**

(1) Take 1 mL of freshly prepared silver nitrate (2%) in a test tube add 1-2 drops of sodium hydroxide solution to it and shake it well, a dark brown ppt. of silver oxide appears. Dissolve the ppt. by adding ammonium hydroxide solution dropwise.

(2) Now, add an aqueous or alcoholic solution of about 50 mg of organic compound.

(3) Heat the reaction mixture on a water bath for 5 minutes.

(4) Formation of a deposit of silver, on the inner side of the test tube indicates the presence of reducing sugars.

II. **Test for starch**

III. **Iodine test**

Heat the small amount of substance with water to get an aqueous colloidal solution of starch. Add few drops of iodine solution. The appearance of blue colour indicates the presence of starch.

IV. **Test for fats**

Add few crystals of dry potassium hydrogen sulphate to 3 mL of mustard oil/ghee taken in a test tube and heat the content of the test tube gently, a pungent smell confirms the presence of oil and fat.

**Test for proteins**

(1) Warm 2-3 mL of egg albumin solution in water with two drops of sodium hydroxide solution after adding a few drops of copper sulphate solution, for about 5 minutes.

(2) Appearance of violet colour indicates the presence of proteins.

(Apppearance of violet colour on account of the formation of complex species by Cu^{2+} ions with - CONH_{2} group confirms the presence of protein in the sample.)
Qualitative Analysis

**Experiment 15: To detect one cation and one anion from the following:**

- **Cations** - Pb\(^{2+}\), Cu\(^{2+}\), As\(^{3+}\), Al\(^{3+}\), Fe\(^{3+}\), Mn\(^{2+}\), Ni\(^{2+}\), Zn\(^{2+}\), Co\(^{2+}\), Ca\(^{2+}\), Sr\(^{2+}\), Ba\(^{2+}\), Mg\(^{2+}\), NH\(_4\)^+
- **Anions** - CO\(_3\)^{2-}, S\(^{2-}\), SO\(_3\)^{2-}, SO\(_4\)^{2-}, NO\(_2\)^-, NO\(_3\)^-, Cl\(-\), Br\(-\), I\(-\), PO\(_4\)^{3-}, CH\(_3\)COO\(^{-}\) (Insoluble salts to be excluded)

**Apparatus:**
Micro test-tubes with stand, test tube holder, kerosene burner, W-tube, well plate, dispensing bottles, china dish, cotton swabs, charcoal cavity, W. Tube etc.

**Chemicals:**
6MH\(_2\)SO\(_4\), 6MHCl, lime water, sodium carbonate, sodium metal, BaCl\(_2\) solution, KMnO\(_4\) solution, KI solution, starch solution, acetic acid, conc. H\(_2\)SO\(_4\), NH\(_4\)OH, MnO\(_2\), Cu turnings, AgNO\(_3\) solution, K\(_2\)Cr\(_2\)O\(_7\), lead acetate solution, CCl\(_4\), ethanol, ammonium molybdate, conc HNO\(_3\), borax, cobalt nitrate, Nessler’s reagent, bromine water, chlorine water, lime water, etc.

**Introduction:**
The inorganic qualitative analysis involves the identification of ions present in a sample. The salt consists of anion and cation. The basis of identification is the observed chemical behaviour of the components. The salt analysis may be systematically carried out in the following three steps:

i) Preliminary examination
ii) Detection of anions and
iii) Detection of cations

**Preliminary examination:**
First we proceed to examine the physical properties of the salt. This is followed by the action of heat on the salts and also certain tests, such as flame test etc.

**Detection of Anions:**
A preliminary hint about some anions may be obtained by the formation of volatile or gaseous products when the salt is treated with
i) dil. H₂SO₄ (for CO₃²⁻, S²⁻, SO₃²⁻, NO₂⁻ and CH₃COO⁻)
ii) conc. H₂SO₄ (for Cl⁻, Br⁻, I⁻ and NO₃⁻)

1. Take about 10 mg of the salt in a dry test tube with the help of a spatula.

2. Heat the test tube over the burner and observe the change. Note the colour and odour of the gases if evolved as given in Table 2.

### Table 2: Action of Heat

<table>
<thead>
<tr>
<th>Observation</th>
<th>Inference</th>
<th>Chemistry Involved</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonia gas evolves</td>
<td>NH₄⁺</td>
<td>(NH₄)₂CO₃ → NH₄⁺CO₃⁻H₂O</td>
</tr>
<tr>
<td>Carbon dioxide evolves</td>
<td>CO₃²⁻ present</td>
<td>CaCO₃ → CaO + CO₂</td>
</tr>
<tr>
<td>Sulphur dioxide evolves</td>
<td>SO₃²⁻ may be present</td>
<td>2FeSO₄ → Fe₂O₃ + SO₂ + SO₃</td>
</tr>
<tr>
<td>Nitrogen dioxide evolves</td>
<td>NO₃⁻</td>
<td>2Cu(NO₃)₂ → 2CuO + 4NO₂ + O₂</td>
</tr>
</tbody>
</table>

**Colour of the salt on heating changes from**

1. Some salts change their colour blue to white
2. Green to yellow
3. White (when cold) to yellow (when hot)

### Flame Test

Prepare a paste of small amount of the salt with one or two drops of conc. HCl on a watch glass. Take the paste on the loop of Nichrome wire or cotton swab, put it in the burner’s flame and observe the colour as given in Table 3.

### Table 3: Flame Test

<table>
<thead>
<tr>
<th>Flame colour observed by naked eye</th>
<th>Inference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deep blue green</td>
<td>Cu²⁺</td>
</tr>
<tr>
<td>Brick red</td>
<td>Ca²⁺</td>
</tr>
<tr>
<td>Apple green</td>
<td>Ba²⁺</td>
</tr>
<tr>
<td>Crimson (deep red)</td>
<td>Sr²⁺</td>
</tr>
</tbody>
</table>

A confirmation is made by other specific tests. Other anions (SO₄²⁻ and PO₄³⁻) are identified by different reagents.

For performing some identification tests for anions, it is essential
to prepare the salt solution. Depending upon the solubility of the
given salt, we may prepare its aqueous solution or water extract or
sodium carbonate extract for carrying out the tests.

**Water Extract (WE):**

Boil a few mg of the salt with 2 mL distilled water for 2-3 minutes

**Sodium Carbonate Extract (SE):**

If the salt is insoluble in water, its sodium carbonate extract is
prepared. On boiling the salt with sodium carbonate solution, double
decomposition takes place resulting in the formation of soluble sodium
salt of the anion. Mix 5 mg of the salt with 15 mg Na₂CO₃ in a boiling
tube. After transferring this mixture add about 10 mL of distilled
water in 25 mL beaker and boil for about 5 min. Filter, the filtrate is
the sodium carbonate extract (SE).

**Detection of Cations:**

All cations may be divided into six groups depending upon the
difference in solubility of their chlorides, sulphides, hydroxides,
carbonates under different conditions of acidity and alkalinity. The
reagent used to test the presence of a group is known as a group
reagent.

The first step before proceeding to systematic wet analysis of
cations is to dissolve the salt in a suitable solvent. The clear solution
is known as original solution (OS).

**Instructions:**

1. Note the colour, odour and state of the given salt as given in the
   Table 1.

<table>
<thead>
<tr>
<th>Physical Properties</th>
<th>Inference</th>
</tr>
</thead>
<tbody>
<tr>
<td>White Colour (colourless)</td>
<td>Pb²⁺, As³⁺, Al³⁺, Zn²⁺, Ca²⁺, Ba²⁺, Sr²⁺, Mg²⁺ or NH₄⁺ salt may be present.</td>
</tr>
<tr>
<td>Bluish or bluish green colour</td>
<td>Cu²⁺ or Ni²⁺ Salt</td>
</tr>
<tr>
<td>Yellowish brown colour</td>
<td>Fe²⁺ or Fe³⁺ Salt</td>
</tr>
<tr>
<td>Smell of ammonia gas</td>
<td>NH₄⁺ Salt</td>
</tr>
<tr>
<td>Smell of hydrogen sulphide gas</td>
<td>S²⁻</td>
</tr>
<tr>
<td>Heavy</td>
<td>Pb²⁺, Ba²⁺ Salt</td>
</tr>
<tr>
<td>Light</td>
<td>Al³⁺, Mg²⁺, Ca²⁺ Salt</td>
</tr>
</tbody>
</table>
Classification of cations into groups: Classification of the cations into groups is based on the values of their solubility products with different anions.

<table>
<thead>
<tr>
<th>Group</th>
<th>Cations</th>
<th>Group Reagent</th>
<th>Precipitated as</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>Pb²⁺, Hg⁺, Ag⁺</td>
<td>Dil HCl</td>
<td>Chlorides - e.g. PbCl₂</td>
</tr>
<tr>
<td>II</td>
<td>Pb²⁺, Cu²⁺, As³⁺</td>
<td>S²⁻ ions in acidic medium</td>
<td>Sulphides - PbS, CuS, As₂S₃</td>
</tr>
<tr>
<td>III</td>
<td>Fe³⁺, Al³⁺</td>
<td>NH₃·OH</td>
<td>Fe(OH)₃, Al(OH)₃</td>
</tr>
<tr>
<td>IV</td>
<td>Ni²⁺, Co²⁺, Zn²⁺, Mn²⁺</td>
<td>S²⁻ ions in basic medium</td>
<td>NiS, CoS, ZnS, MnS</td>
</tr>
<tr>
<td>V</td>
<td>Ca²⁺, Ba²⁺, Sr²⁺</td>
<td>(NH₄)₂CO₃</td>
<td>CaCO₃, BaCO₃, SrCO₃</td>
</tr>
<tr>
<td>VI</td>
<td>Mg²⁺, NH₄⁺,NH₄⁺⁺, K⁺</td>
<td>Individual tests</td>
<td></td>
</tr>
</tbody>
</table>

Group VI cations are tested individually with different reagents.

Anion analysis

Anions are classified as follows:

- **Dil. H₂SO₄ group**
  - CO₃²⁻, NO₃⁻, S²⁻, SO₃²⁻, CH₃COO⁻
- **Conc. H₂SO₄ group**
  - F⁻, Cl⁻, Br⁻, I⁻, NO₃⁻
- **Independent radicals**
  - SO₄²⁻, PO₄³⁻

**Dil. H₂SO₄ Group**:

Take a pinch of salt in a semi micro test tube, add few drops of dil H₂SO₄ and heat. Look for anyone of the following observations:

2. Colourless gas with rotten egg smell - S²⁻ indicated
3. Colourless gas with pungent smell - SO₃²⁻ indicated. Use W-tube for SO₂ test using dil solution of potassium permanganate or dichromate solution.
4. Brown fumes - NO₂⁻ indicated
5. Vinegar smell - CH₃COO⁻ indicated

If anyone of the above is detected, carry out the confirmatory wet test as follows:
1. **Confirmatory test for CO$_3^{2-}$ ion** - Take a pinch of salt in one arm of W-tube and transfer two drops of lime water in the other arm with the help of dropper. Now transfer 2-3 drops of dil HCl with the help of a dropper in the first arm. The gas from this arm bubbles through the arm containing lime water. Lime water turns milky - CO$_3^{2-}$ confirmed.

2. **Confirmatory test for S$_2^-$ ion** - Take few drops of OS in a well in the well plate and add a drop of sodium nitroprusside solution - purple or violet colour confirms S$_2^-$.

3. **Confirmatory test for SO$_3^{2-}$ ion** - Take 4-5 drops of OS and add a few drops of BaCl$_2$ solution – white ppt soluble in dil HCl confirms SO$_3^{2-}$.

4. **Confirmatory test for NO$_2^-$ ion** - (i) Take a few drops of OS in a well in the well plate, add a drop of dil acetic acid followed by a few drops of ferrous sulphate solution — dark brown or black colour confirms NO$_2^-$. (ii) Take 5 mg of solid salt in a micro test-tube and add 3 drops of acetic acid and 5 mg of KI. Warm and shake with 5-6 drops of carbon tetrachloride or chloroform. Violet colour due to iodine in the carbon tetrachloride or chloroform layer confirms presence of nitrite ions. This test can also be done for iodide ions.

5. **Confirmatory test for CH$_3$COO$^-$**
Rub a pinch of the salt with oxalic acid-vinegar smell confirms acetate ion.

**Conc. H$_2$SO$_4$ group**

Take a spatula of salt (10-15 mg) in a micro test tube, add few drops of conc. H$_2$SO$_4$ and heat. Look for anyone of the following observations or use W-tube for halide ions test taking AgNO$_3$ solution in one arm.

1. Colourless gas with pungent smell, gives dense white fumes when a glass rod dipped in NH$_4$OH is brought near the mouth of test tube - Cl$^-$ indicated.

2. Reddish brown vapours with pungent smell, turn starch paper yellow and do not intensify on adding Cu turning - Br$^-$ indicated.

3. Violet vapours, turn starch solution/paper black - I$^-$ indicated.

4. Brown fumes with pungent smell, intensify on adding a Cu turning - NO$_3^-$ indicated.
If anyone of the above is detected carry out the confirmatory wet test as follows:

1. **Confirmatory test for NO₃⁻ ion** – Transfer one drop of salt solution in one of the wells of well plate and add one drop of freshly prepared FeSO₄ solution. Add 3 drops of conc. H₂SO₄ slowly from the side of the well – a brown ring at the junction of the liquids confirms Cl⁻.

2. **Confirmatory test for Cl⁻ ion** – Transfer a small pinch of salt in one of the arms of W-tube and add a pinch of solid K₂Cr₂O₇ followed by 2-3 drops of conc. H₂SO₄. Transfer 2-3 drops of NaOH solution in the other arm of W-tube. Heat carefully the first arm of the W-tube by holding it by the holder. Now take one drop of this NaOH (yellow in colour) in a well, add a drop of dil acetic acid followed by 2 drops of lead acetate solution – Yellow ppt confirms Cl⁻.

3. **Confirmatory test for Br⁻ ion** – Transfer 3-4 drops of OS in one arm of the W-tube and 2 drops of silver nitrate solution in the other arm. Transfer a few drops of dil. HNO₃ in the first arm with the help of a dropper and warm. Light yellow ppt partially soluble in NH₄OH solution confirms Br⁻.

4. **Confirmatory test for I⁻ ion** – Repeat the above test with salt solution – Yellow ppt insoluble in NH₄OH solution confirms I⁻.

**Independent radicals:**

1. **SO₄²⁻** Boil a pinch of salt with small amount of dil. HCl (filter if needed to remove any solid present) and add BaCl₂ solution – White ppt insoluble in conc. HCl confirms SO₄²⁻.

2. **PO₄³⁻** Boil a pinch of salt with few drops of conc. HNO₃ and add ammonium molybdate solution in slight excess – A yellow ppt confirms PO₄³⁻.

**Scheme of classification of cations:**

**Original solution (OS):** Take a microspatula full of salt in a micro test tube and add 2 mL water. Shake to dissolve the salt. If not soluble, add 2-3 drops of dil. HCl. Warm if necessary.
**GROUP I**

<table>
<thead>
<tr>
<th>Ppt-Gp I present</th>
<th>Pb²⁺ Present</th>
<th>Test for Pb²⁺ in hot solution</th>
<th>Proceed to analyse and confirm Gp I radicals</th>
</tr>
</thead>
<tbody>
<tr>
<td>Black or yellow ppt-</td>
<td>Black ppt-</td>
<td></td>
<td>Proceed to analyse and confirm Gp II radicals</td>
</tr>
<tr>
<td>Gp II present</td>
<td>Pb²⁺ or Cu²⁺</td>
<td>Yellow ppt-</td>
<td>As³⁺ present</td>
</tr>
<tr>
<td>BROWN or white ppt-Gp III present</td>
<td>Brown (Fe³⁺) white gelatinous (Al³⁺)</td>
<td></td>
<td>Proceed to analyse and confirm Gp III radicals</td>
</tr>
<tr>
<td>Black ppt-Ni²⁺, Co²⁺</td>
<td>Black ppt-</td>
<td></td>
<td>Proceed to analyse and confirm Gp IV radicals</td>
</tr>
<tr>
<td>Gp V present</td>
<td>White ppt-</td>
<td></td>
<td>Proceed to analyse and confirm Gp V</td>
</tr>
<tr>
<td>Ca²⁺, Ba²⁺, Sr²⁺</td>
<td></td>
<td></td>
<td>No ppt-Gp VI present</td>
</tr>
</tbody>
</table>

Separate the white ppt and wash with water. Boil the ppt with 1/2 mL water and transfer 2 drops each of the resulting clear solution into two wells in the well plate.

To one well, add one drop of potassium iodide solution—
Yellow ppt confirms Pb²⁺

To the other well, add one drop of potassium chromate solution—
Yellow ppt confirms Pb²⁺
GROUP II

<table>
<thead>
<tr>
<th>Yellow ppt As(^{3+}) present</th>
<th>Black ppt-Pb(^{2+}) or Cu(^{2+}) present</th>
</tr>
</thead>
<tbody>
<tr>
<td>Confirmation</td>
<td></td>
</tr>
<tr>
<td>Dissolve the ppt in min</td>
<td>To the other part of solution add</td>
</tr>
<tr>
<td>quantity of conc HNO(_3)</td>
<td>NH(_4)OH in excess</td>
</tr>
<tr>
<td>Add ammonium Molybdate solution and heat</td>
<td>Blue coloured solution Cu(^{2+}) indicated.</td>
</tr>
<tr>
<td>Yellow ppt confirms As(^{3+})</td>
<td></td>
</tr>
<tr>
<td>Confirmation</td>
<td></td>
</tr>
<tr>
<td>Transfer 1 drop of OS in well</td>
<td>Transfer 2 drops of blue solution to two</td>
</tr>
<tr>
<td>plate and add 1 drop dil H(_2)SO(_4)</td>
<td>wells in the well plate:</td>
</tr>
<tr>
<td>and 1 drop of alcohol White ppt-</td>
<td>1. To one well, add a drop of acetic acid</td>
</tr>
<tr>
<td>Pb(^{2+}) indicated.</td>
<td>followed by a drop of potassium ferrocyanide solution.</td>
</tr>
<tr>
<td>Do other test for Pb(^{2+}) as</td>
<td>Chocolate brown ppt-Cu(^{2+}) confirmed.</td>
</tr>
<tr>
<td>given in Group I table</td>
<td>2. To the other well, add a drop of acetic acid followed by a drop of potassium iodide solution.</td>
</tr>
<tr>
<td></td>
<td>Dirty yellow ppt-Cu(^{2+}) confirmed.</td>
</tr>
<tr>
<td></td>
<td>Add 3 drops of CCl(_4)/CHCl(_3) and</td>
</tr>
<tr>
<td></td>
<td>shake, violet colour in organic layer-Cu(^{2+}) confirmed.</td>
</tr>
</tbody>
</table>

GROUP III

Transfer 4 drops of OS in a micro test-tube and add a pinch of ammonium chloride followed by 2 drops of ammonium hydroxide.

**Confirmation**

1. Brown ppt. Transfer 2 drops of OS to a well in the well plate and add a drop of potassium ferrocyanide solution. Prussian blue colour-Cu\(^{2+}\) confirmed.

Transfer one drop of OS in well plate and add 1 drop of KSCN/NH\(_4\)CN-Blood red colour-Cu\(^{2+}\) confirmed.
Co\(^{2+}\) indicated

 Confirmation-
 Transfer 2–3
drops of OS and a
crystal of
ammonium thiocyanate-
Blue colour in the
either layer
 confirms Co\(^{2+}\).
 Transfer 2 drops
of OS in well plate
and add 2 drops
of NH\(_4\)SCN and
 amyl alcohol-
blue colour in amyl
 alcohol layer.

Ni\(^{2+}\) indicated

 Confirmation-
 Transfer 2
drops of OS in a
 well of well
 plate, add a
drop of dimethyl
 glyoxime
solution and
NH\(_4\)OH Pink ppt
confirms Ni\(^{2+}\).

Zn\(^{2+}\) indicated

 Confirmation-
 Transfer 3
drops of OS to a
well and add
NaOH dropwise-
white ppt soluble
in excess NaOH-
Zn\(^{2+}\) confirmed.
 Transfer 2 drops
of solution to a
well and add a
drop of
potassium ferrocyanide-
bluish white ppt-
Zn\(^{2+}\) confirmed.

Mn\(^{2+}\) indicated

 Confirmation-
 Transfer 2 drops
of OS to a well
and add NaOH
 drop wise-white
ppt which on
addition of Br\(_2\)
water turns
black or changes
without Br\(_2\), the
ppt drops of dil
HCl and colour
on standing for
some time.
Mn\(^{2+}\) confirmed.

White ppt indicates Ba\(^{2+}\) or Sr\(^{2+}\) or Ca\(^{2+}\).
Separate the ppt and wash with water. Dissolve in hot dil acetic acid and divide
the solution into three parts.

Confirmation to be carried out in the sequence given below.

### GROUP IV

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Co(^{2+})</td>
<td>Ni(^{2+})</td>
<td>Zn(^{2+})</td>
</tr>
<tr>
<td>Confirmation-</td>
<td>Confirmation-</td>
<td>Confirmation-</td>
</tr>
<tr>
<td>Transfer 2–3</td>
<td>Transfer 2</td>
<td>Transfer 3</td>
</tr>
<tr>
<td>drops of OS</td>
<td>drops of OS</td>
<td>drops of OS</td>
</tr>
<tr>
<td>and a crystal</td>
<td>in a well of</td>
<td>to a well</td>
</tr>
<tr>
<td>of ammonium</td>
<td>well plate</td>
<td>and add NaOH</td>
</tr>
<tr>
<td>thiocyanate-</td>
<td>add a drop of</td>
<td>dropwise-</td>
</tr>
<tr>
<td>Blue colour</td>
<td>dimethyl glyoxime</td>
<td>white ppt</td>
</tr>
<tr>
<td>in the either</td>
<td>solution and</td>
<td>soluble in</td>
</tr>
<tr>
<td>layer confirms</td>
<td>NH(_4)OH Pink</td>
<td>excess NaOH</td>
</tr>
<tr>
<td>Co(^{2+}).</td>
<td>ppt confirms Ni</td>
<td>Zn(^{2+}) confirmed.</td>
</tr>
<tr>
<td>Transfer 2</td>
<td>Transfer 2</td>
<td></td>
</tr>
<tr>
<td>drops of OS</td>
<td>drops of OS</td>
<td></td>
</tr>
<tr>
<td>in well plate</td>
<td>in well plate</td>
<td></td>
</tr>
<tr>
<td>and add 2 drops</td>
<td>add a drop of</td>
<td>and add a drop</td>
</tr>
<tr>
<td>of NH(_4)SCN</td>
<td>dimethyl glyoxime</td>
<td>of solution to</td>
</tr>
<tr>
<td>and amyl</td>
<td></td>
<td>a well and add</td>
</tr>
<tr>
<td>alcohol-blue</td>
<td></td>
<td>a drop of</td>
</tr>
<tr>
<td>colour in amyl</td>
<td></td>
<td>potassium</td>
</tr>
<tr>
<td>alcohol layer.</td>
<td></td>
<td>ferrocyanide-</td>
</tr>
<tr>
<td></td>
<td></td>
<td>bluish white</td>
</tr>
<tr>
<td></td>
<td></td>
<td>ppt-Zn(^{2+}) confirmed.</td>
</tr>
</tbody>
</table>

### GROUP V

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Ba(^{2+})</td>
<td>Sr(^{2+})</td>
<td>Ca(^{2+})</td>
</tr>
<tr>
<td>1. Transfer 2</td>
<td>1. To a part of</td>
<td>1. To a part of</td>
</tr>
<tr>
<td>drops of</td>
<td>the solution</td>
<td>solution add</td>
</tr>
<tr>
<td>solution to a</td>
<td>and add 2 drops</td>
<td>2 drops of</td>
</tr>
<tr>
<td>well and add</td>
<td>of ammonium</td>
<td>ammonium oxalate</td>
</tr>
<tr>
<td>2 drops of</td>
<td>sulphate solution</td>
<td>solution in a</td>
</tr>
<tr>
<td>potassium</td>
<td>in as micro</td>
<td>test tube and</td>
</tr>
<tr>
<td>chromate</td>
<td>test tube and</td>
<td>warm-White ppt</td>
</tr>
<tr>
<td>solution-Yellow</td>
<td>warm-White ppt</td>
<td>confirms Sr(^{2+})</td>
</tr>
<tr>
<td>ppt confirms</td>
<td>confirms Sr</td>
<td>2. Perform flame test-dip</td>
</tr>
<tr>
<td>Ba(^{2+})</td>
<td>(^{2+})</td>
<td>a cotton swab</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(ear bud) in</td>
</tr>
<tr>
<td></td>
<td></td>
<td>water and touch</td>
</tr>
<tr>
<td></td>
<td></td>
<td>it to the salt.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Heat in the</td>
</tr>
<tr>
<td></td>
<td></td>
<td>reducing flame</td>
</tr>
<tr>
<td></td>
<td></td>
<td>and observe it</td>
</tr>
<tr>
<td></td>
<td></td>
<td>immediately-</td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>Apple green</strong></td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>flame</strong></td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>confirms Ba</strong></td>
</tr>
<tr>
<td></td>
<td></td>
<td>(^{2+})</td>
</tr>
</tbody>
</table>

Ca\(^{2+}\) 1. To a part of solution add 2 drops of ammonium oxalate solution in a test tube. Add few drops of NH\(_4\)OH and scratch the side of test tube-White ppt confirms Ca\(^{2+}\).
2. Perform flame test-dip a cotton swab (ear bud) in water and touch it to the salt. Heat it in the reducing flame-**Brick red flame confirms Ca**\(^{2+}\).
Comments

The following points should be kept in mind while using micro kit for qualitative analysis:

1. Solution (original) (OS) should be prepared in micro test tubes.
2. Whenever heating is required, prefer test tubes over well plates.
3. For confirmatory tests, prefer well plates.
4. For all gas confirmation, use W- Tube e.g. CO$_3^{2-}$, NO$_2^-$, etc.

**Group VI**

<table>
<thead>
<tr>
<th></th>
<th>Mg$^{2+}$</th>
<th>NH$_4^+$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Transfer 6–7 drops of OS in a micro test-tube add a pinch of solid NH$_4$Cl, shake and add NH$_4$OH in slight excess. Add few drops of ammonium phosphate solution (or any other soluble or ammonium hydrogen phosphate) and rub the side of the test tube with a glass rod.</td>
<td>1. Take one micro spatula full of salt in a micro test tube and add few drops of strong solution of NaOH, heat – Gas smelling of NH$_3$ indicates H$_4^-$ ion.</td>
<td></td>
</tr>
<tr>
<td><strong>A white ppt confirms Mg$^{2+}$</strong></td>
<td>2. Suck the gas evolved in the above test using a dropper. Bubble the gas into drops of Nessler’s reagent taken in a well of well plate.</td>
<td><strong>Brown ppt confirms NH$_4^+$</strong></td>
</tr>
</tbody>
</table>

| If no ppt is formed in Gp V test, Mg$^{2+}$ or NH$_4^+$ may be present |
NOTES