DEEPSHIKHA KALA SANSTHAN

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ESTD. 1976

DEEPSHIKHA

COLLEGE OF TECHNICAL EDUCATION

CHEMISTRY MANUAL

Building Careers, Building the Nation

Chemistry Laboratory

The Department of Chemistry offers practical training to students enrolled in the Science programme, with Chemistry as a subsidiary subject for those who have chosen Physics as their major discipline. The college has two well-equipped Chemistry laboratories designed to provide an effective and safe environment for practical learning.

Each laboratory is furnished with adequate equipment, chemicals, glassware, and other essential facilities required to conduct undergraduate practical experiments. The laboratories also include a **Chemical Stock Room**, **Instrumentation Room**, and **Balance Room**, ensuring the proper storage, maintenance, and handling of laboratory materials and instruments.

To promote systematic laboratory work and individual responsibility, each student is allotted a personal locker containing the necessary laboratory glassware and accessories for performing practical experiments.

The Chemistry laboratories are equipped with modern instruments and infrastructure that support experiential learning, scientific investigation, and skill development. Students gain hands-on experience through carefully designed experiments, enabling them to understand theoretical concepts, develop analytical thinking, and acquire practical laboratory techniques in accordance with the curriculum.

The department strictly adheres to laboratory safety protocols and promotes good laboratory practices, ensuring a secure and productive learning environment for all students.

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GUIDELINES FOR CARRYING OUT EXPERIMENTS

LAB RULES

1. Bring the laboratory manual and a laboratory notebook to every scheduled laboratory session.
2. Careful notes should be taken during each laboratory lecture. The instructor will generally provide information on the chemistry underlying the project, as well as advice on the techniques that you will use. Some of this information should be included in your laboratory report.
3. You may work in the Chemistry UG laboratories only during your regularly scheduled laboratory period and only when class is in session.
4. If you miss a laboratory for a legitimate reason, you must obtain permission from your regular laboratory instructor to make-up the lab at another time. Make up labs are only allowed when space allows and with the approval of the host professor. During the make-up laboratory, you must move your equipment to an unoccupied lab bench.
5. During the first scheduled laboratory period, you will be assigned a laboratory locker to which you alone will have the combination. The locker equipment is your responsibility while you are in the course and you should be diligent to keep the equipment clean.
6. Test tube brushes and soap solutions are available at each sink.
7. The Chemistry Department maintains a stockroom to store the chemical and apparatus. This stockroom is open during all scheduled laboratory periods for the acquisition of equipment necessary to replace broken items.

LAB SAFETY

TRAINING AND SAFETY RULES

- Attendance is required at a presentation on laboratory safety that will be shown during your first scheduled laboratory. Each student must give the instructor a signed form indicating that the presentation was attended and that any associated training materials were examined.
- The laboratory is equipped with a fire blanket, showers, eye wash, and first aid supplies.
- Learn the locations and proper use of these items.

At all times when you are working in the chemistry laboratory you should use prudent practices. Recognize that safety is, ultimately, everyone's individual responsibility. Never work alone in any laboratory.

Avoid the most common causes of accidents

- Exercise care when picking up potentially hot objects.
- Insert glass objects into rubber stoppers and corks with extreme care.

Avoid contact with laboratory chemicals

- Wear clothing that protects as much of your body as possible. Closed-toe shoes are required. All skin below the waist must be covered.
- Use department-approved eye-protection at all times.
- Keep the laboratory bench and work area orderly, clean, and free of items not related to

the experiment at all times. Specifically, electronic devices are not allowed on the bench.

- Never sit on or lean against the laboratory bench.
- Use a fume hood when directed to do so.
- Food or drink should only be consumed in the lecture area of the room. Do not chew gum during laboratory sessions.
- Dispose of waste materials and excess chemicals in the appropriate containers as indicated by your instructor.

When Emergencies do Occur

- Always keep in mind that the first response to the exposure of the eyes or skin to a chemical is immediate, thorough irrigation with water.
- Report all accidents, however minor, to the laboratory instructor immediately.
- Know the exact location of all safety equipment and how to use it.

Preparation is important

- Perform only assigned experiments. Do not attempt to modify the written procedures unless instructed to do so.
- When conducting experiments ask yourself, "What are the worst possible things that could go wrong?" and "How will I deal with them?" Don't do the experiment until you are certain of your answers.
- Read the label on the container to be certain it contains the required chemical.

FIRST AID & EMERGENCY MEASURES

- **Acid on clothing:** Neutralize with dilute ammonium hydroxide. Then wash thoroughly with water
- **Alkali on clothing:** Neutralize with dilute acetic acid. Then wash thoroughly with water.
- **Acid in eye:** Wash thoroughly and profusely with running water. Bathe the eye with a 2 % sodium bicarbonate solution, using an eye cup. Dry with sterile gauze and put several drops of olive oil into the eye.
- **Alkali in eye:** Wash thoroughly and profusely with running water and the eyelids should be held widely open especially when caustic alkalies have entered the eye. Bathe with boric acid solution. Dry and add a drop of olive oil into the eye.
- **Ordinary heat burns:** Don't use water. Apply sodium bicarbonate, Vaseline paste or burnol and consult a doctor
- **Acid burns:** Wash first with running water and then with sodium bicarbonate solution. Cover with solid sodium bicarbonate for 10 minutes. Wash off and apply carron oil (a mixture of equal parts of lime water and linseed oil).
- **Alkali burns:** Wash first with running water and then with boric acid solution. Cover with powdered boric acid for 10 minutes. Wash off and apply sodium bicarbonate Vaseline paste.
- **Cuts:** Remove particles of glass, if any are present. Wash the wound with water.
- Then apply tincture of iodine and cover with a sterile bandage
- Poisoning by strong acid: Give plenty of water or milk. Then give two tablespoons of lime water
- Poisoning by caustic alkali's: Give plenty of water or milk. Then give orange or lemon juice

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- Fire: Extinguish gas burners in the vicinity. Use buckets of dry sand/fire-extinguishers to cease the fire.

To ensure a safe and productive laboratory environment, all students, faculty members, and laboratory staff must strictly adhere to the following safety rules:

General Safety Rules

1. Always wear a laboratory coat while performing practical work.
2. Wear laboratory safety glasses or chemical splash goggles whenever chemicals are handled or stored, and whenever there is a risk of splashes or flying particles.
3. Avoid all unnecessary skin and eye contact with chemicals.
4. Minimize exposure to chemicals by handling them carefully and following the prescribed procedures.
5. Never taste chemicals or intentionally inhale their vapours.
6. Never consume food or beverages, chew gum, smoke, or apply cosmetics in the laboratory.
7. Long hair must be tied back, and loose clothing, scarves, or jewellery must be secured to prevent accidental contact with chemicals or equipment.
8. Always keep your work area clean, organized, and free from unnecessary materials.
9. Do not sit or lean on laboratory benches.
10. Keep laboratory aisles and floors clear of bags, spilled liquids, ice, broken glass, or any objects that may cause slips or falls.

Laboratory Conduct

11. Follow all instructions provided by the laboratory instructor or technician.
12. Never perform unauthorized experiments or modify the prescribed procedure without permission.
13. Do not run, engage in horseplay, practical jokes, or any disruptive behaviour inside the laboratory.
14. The use of personal audio or video devices, except for academic purposes with permission, is prohibited.
15. Never work in the laboratory without the supervision of the laboratory instructor or authorized staff.
16. Be familiar with the location and proper use of emergency exits, fire extinguishers, first-aid kits, eye-wash stations, safety showers, and other emergency equipment.

Safe Handling of Equipment

17. Never attempt to catch a falling glass object or chemical container.
18. Never point the open end of a test tube containing chemicals toward yourself or others.
19. Handle all glassware and laboratory instruments carefully to prevent breakage and injury.
20. Report any damaged equipment, broken glassware, chemical spills, or accidents immediately to the laboratory instructor.

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Fire and Heating Safety

21. Never leave a Bunsen burner or any heating apparatus unattended while in use.
22. Ensure that no flammable chemicals or solvents are present near an open flame.
23. Turn off all burners, gas valves, electrical equipment, heating devices, and water taps immediately after completing the experiment.
24. Dispose of chemical waste only in the designated waste containers as instructed by the laboratory staff.

Emergency Procedures

25. Immediately report all accidents, injuries, chemical spills, or unsafe conditions to the laboratory instructor.
26. Follow the emergency evacuation procedure whenever instructed.
27. In case of chemical exposure, use the eye-wash station or safety shower immediately and seek medical assistance.
28. Always maintain discipline and cooperate with laboratory personnel during emergencies.

Remember

Safety is everyone's responsibility. Careful laboratory practices protect you, your classmates, and the laboratory environment. Strict compliance with these rules is mandatory for all students and staff.

LABORATORY NOTEBOOK GUIDELINES

Maintaining an accurate and well-organized laboratory notebook is an essential part of scientific learning and professional laboratory practice. The laboratory notebook serves as a permanent record of all experiments performed, observations made, and results obtained during practical sessions.

Laboratory reports and practical assessments are based on the information recorded in the notebook. Therefore, students are expected to record all observations and data carefully, accurately, and systematically. Complete and reliable records also help explain unexpected results, identify experimental errors, and provide valuable references for future study or research.

In scientific research and professional laboratories, laboratory notebooks are considered official records and may be referred to months or even years after an experiment has been completed. For this reason, all entries should be clear, complete, and permanently recorded.

Notebook Requirements

Students are required to maintain a **hard-cover, bound laboratory notebook** throughout the course. The following guidelines must be strictly followed:

1. Record all experimental observations and data directly in the laboratory notebook using **permanent blue or black ink**.
2. Do not record observations on loose sheets of paper or any temporary medium for later copying.
3. Each laboratory session must begin with the **date**, **experiment title**, and **objective** clearly

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written.

4. Record observations, measurements, and calculations immediately as they are obtained.
5. Do not erase mistakes. Any incorrect entry should be crossed out with a **single line** so that the original entry remains legible.
6. Never remove or tear pages from the laboratory notebook.
7. Maintain neat, legible, and well-organized records so that another person can easily understand and reproduce the experiment.
8. Use properly labelled tables wherever possible to organize experimental data.

Information to be Recorded

The laboratory notebook should include the following information for every experiment:

- Date of the experiment
- Title and objective of the experiment
- Apparatus and chemicals used (where applicable)
- Experimental procedure (brief stepwise record of the work actually performed)
- Raw experimental data, including measurements such as masses, volumes, temperatures, burette readings, pH values, and other observations
- Significant observations, including colour changes, precipitate formation, gas evolution, phase changes, or any unusual occurrences
- Calculations performed during and after the experiment
- Results obtained
- Precautions followed during the experiment
- Sources of error (if any)
- Final conclusion

Good Laboratory Practice

- Record information immediately; never rely on memory.
- Ensure all entries are accurate, honest, and complete.
- Do not overwrite, alter, or fabricate experimental observations.
- Keep the notebook clean, protected, and available for inspection by the laboratory instructor whenever required.

RECORDING SIGNIFICANT FIGURES IN YOUR NOTEBOOK

- When recording a numeric measurement, the number of significant figures conveys how accurate the measurement is.
- When massing, record all digits on the balance. The last place has uncertainty in it (you may even see the digit change).

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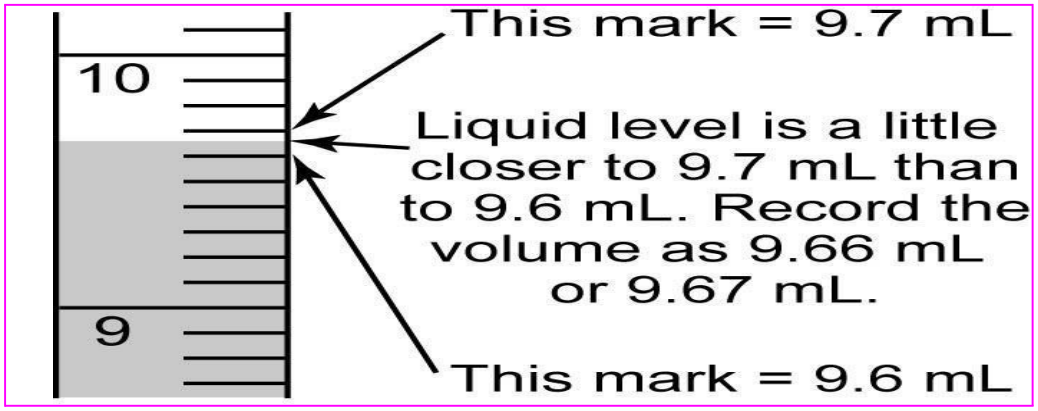
- When measuring volumes using graduated glassware, estimate and record one digit beyond the markings, as shown below in the figure.



EFFECTIVELY COMMUNICATING THROUGH A LABORATORY REPORT

Writing is ubiquitous in the sciences, and students primarily develop their writing skills through writing laboratory reports. As a scientist-in-training, you ultimately need to learn how to make a reasoned and articulate argument that is persuasive and based on scientific evidence; the foundation of that argument needs to be grounded in data that are presented in an organized and thorough manner. The laboratory report communicates to others the results and conclusions you obtained in performing an experiment. You should explain why the experiment was performed, how the experiment was performed, what results were obtained, how the results were analyzed, and what conclusions were reached.

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GENERAL LABORATORY SAFETY PRECAUTIONS

To ensure personal safety and prevent laboratory accidents, every student must observe the following precautions:

1. Be aware of the most common causes of laboratory accidents and take appropriate preventive measures at all times.
2. Handle all laboratory equipment and chemicals with care and follow the instructions of the laboratory instructor.
3. Exercise extreme caution when touching or moving objects that may be hot, including glassware, metal apparatus, hot plates, and heating equipment.
4. Allow heated equipment and glassware to cool before handling, unless proper protective tools are used.
5. Insert glass tubing, thermometers, or other glass objects into rubber stoppers or corks with great care to prevent breakage and injury. Lubricate the glass when appropriate and protect your hands during insertion.
6. Avoid direct contact with laboratory chemicals. Never touch, taste, or intentionally inhale chemicals unless specifically instructed.
7. Wear appropriate laboratory clothing that provides maximum body protection. Loose clothing, scarves, and dangling accessories should be secured before beginning laboratory work.
8. Closed-toe shoes are mandatory. Sandals, slippers, open-toe footwear, or high-heeled shoes are not permitted inside the laboratory.
9. Ensure that all skin below the waist is completely covered by appropriate clothing while working in the laboratory.
10. Wear department-approved safety goggles or protective eyewear at all times while performing

laboratory experiments or whenever chemicals, glassware, or hazardous materials are being handled.

11. Report any accident, injury, broken glassware, or chemical spill immediately to the laboratory instructor.
12. Follow all laboratory safety instructions and emergency procedures to maintain a safe and productive working environment.

VOLUMETRIC ANALYSIS

Volumetric analysis is a widely used quantitative analytical method. As the name implies, this method involves the measurement of volume.

VOLUMETRIC PROCEDURE

[1] A solution is prepared from an accurately weighed sample of the material to be analyzed.

[2] A substance is chosen that will react rapidly and completely with the constituent that is to be determined. A standard solution of this substance is prepared. A standard solution is one of accurately known concentration, usually expressed as molarity with a precision of ± 0.0001 M.

[3] Some of the standard solution is poured into a burette. The burette is graduated (usually in tenths of a mL) so that the volume of solution that passes through the stopcock may be accurately measured.

[4] Standard solution is added slowly from a burette to the "unknown" solution, allowing the reaction to occur. This process is called titration, and the solution in the burette is known as the titrant. Ideally, the titration is continued until the reaction is complete; that is, until the amount of reactant added is exactly the amount required to react with the entire constituent that is being determined. This point is called the equivalence point. The equivalence point is detected by adding an indicator to the "unknown" solution before the titration is begun. An indicator is a substance that gives a color change at or near to the equivalence point. The point at which this change occurs is called the endpoint. The particular indicator that is used depends on the specific reaction involved. The titration is stopped when the endpoint is reached.

[5] The exact volume of standard solution required can be measured, from burette readings before and after the titration. Since the molarity of the standard solution is known, the number of moles of titrant can be calculated. Furthermore, from knowledge of the equation for the reaction, the number of moles of constituents present in the sample can also be calculated.

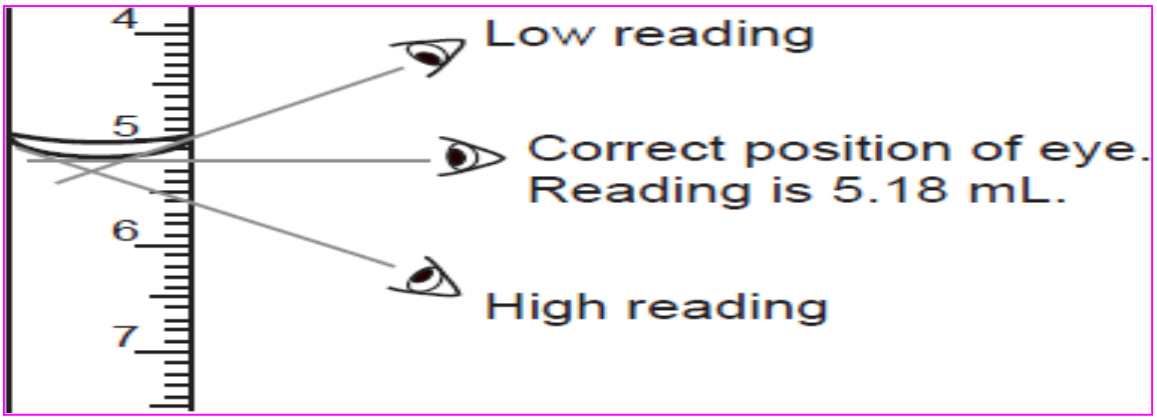
STANDARD SOLUTION

The most accurate and convenient way of preparing a standard solution is to weigh the reagent accurately, dissolve it, and dilute the solution to a definite volume in a volumetric flask. This method can be employed only if the reagent is a primary standard. In order to qualify as a primary standard, a substance must meet the following requirements: it must be obtainable in pure form; it must be stable both in pure form and in solution; it must be easy to dry and keep

dry; and it must be soluble in a suitable solvent. Unfortunately, many useful reagents do not meet these requirements. In such a case, the reagent is dissolved and made up approximately to the concentration desired. This solution is then standardized by titrating it against a primary standard. A solution standardized in this fashion is a secondary standard.

TITRATION PROCEDURE

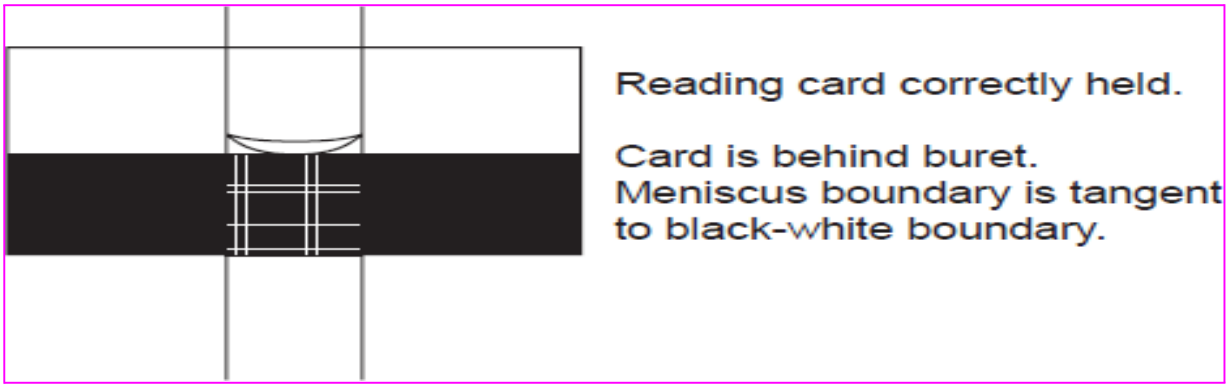
1. Carefully clean the burette with alconox solution and a brush to remove all dirt and grease. Rinse the burette with several portions of tap water by partially filling it, draining a small portion through the tip, and pouring the bulk of the rinse from the top of the burette while rotating it. If any drops of water collect on the walls when the rinse is poured from the burette, the cleaning is unsatisfactory and must be repeated. Finally, rinse the burette with two or three portions of deionized water.
2. Before filling the burette, rinse it with titrant solution 2-3 times, using about 10 mL portions.
3. Place the burette in a burette clamp attached to a large ring stand. Using a funnel, fill the burette with titrant to a level above the zero mark. Place a beaker under the burette and open the stopcock for a few seconds to remove all air from the tip. The top of the solution should now be below the zero mark.
4. Read the burette to ± 0.01 mL. (Because the burette is graduated to 0.1 mL, the second decimal place must be estimated.) To make this reading, it is necessary to locate the meniscus (that is, the surface of the liquid) with respect to the markings. The reading is considerably affected by the position of the eye and by the color of objects behind the burette. The variation of the apparent position of the meniscus is called parallax. To minimize errors due to parallax, the meniscus should be level with the eye (see the Figure given below).



To avoid measurement errors, always read the burette at **eye level** to eliminate parallax error. The bottom of the meniscus should be aligned with the graduation mark when reading colourless solutions. To improve the visibility of the meniscus, place a **white card with a horizontal black strip** behind the burette at the level of the liquid. This provides a clear contrast and enables a more accurate reading.

When using **highly coloured solutions**, the bottom of the meniscus may not be clearly visible. In such

cases, the **top of the meniscus** should be used for taking the burette reading. Accurate eye positioning and proper meniscus reading are essential for obtaining precise and reliable experimental results.



- Place the solution that is to be titrated in a conical flask and add the appropriate indicator. Position the flask under the burette. Add the titrant slowly from the burette while swirling the contents of the flask to assure adequate mixing. As the endpoint is approached, the titrant must be added very slowly-a drop at a time. Usually there is warning as the endpoint is approached. If the endpoint is a color change, the change is produced momentarily where the reagent drops into the solution, but fades with stirring into the bulk of solution. This fading occurs more slowly as the endpoint is approached.
- If the indicator change is a very sharp one, it may be desirable to add standard solution only a half drop at a time near the endpoint. This may be done by opening the stopcock slightly until a drop begins to form on the burette tip. When the droplet has grown to a few hundredths of a mL (one drop is about 0.05 mL), it is touched to the side of the titration vessel and rinsed down with a Little deionized water from the wash bottle.
- If an endpoint is not distinct, or if it is unfamiliar, it may be difficult to decide when the endpoint has actually been reached. In this case, record the burette reading, add another drop, and note the change produced. If the observer is still uncertain, another reading should be recorded, and another drop added. When a series of such readings have been recorded it is easier to select the endpoint in retrospect than by direct approach.
- When the endpoint has been reached, subtract the initial burette reading (step 4 above) from the final reading to obtain the volume of titrant used.

INSTRUMENTATION

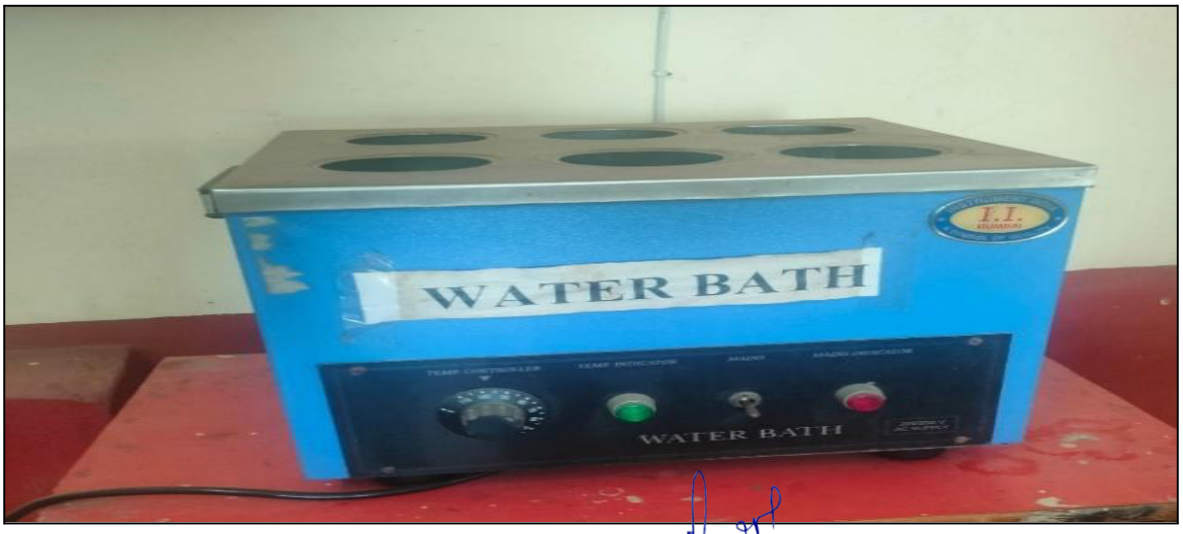
HOT AIR OVEN

Hot air ovens use extremely high temperatures over several hours to destroy microorganisms and bacterial spores. The ovens use conduction to sterilize items by heating the outside surfaces of the item, which then absorbs the heat and moves it towards the center of the item. A hot air oven is widely used in the medical industry to sterilize the equipment and other materials that are used in a laboratory. It is used for delivering the heat treatment to the product. They do not require water and there is not much pressure built up within the oven, unlike an autoclave, making them safer to

work with. This also makes them more suitable to be used in a laboratory environment. They are much smaller than autoclaves but can still be as effective. Items that are sterilized in a hot air oven include: Glassware (like petri dishes, flasks, pipettes, and test tubes), powders (like starch, zinc oxide, and sulfadiazine), materials that contain oils and metal equipment (like scalpels, scissors, and blades). Place the articles at sufficient distances so as to allow free circulation of air in between them and to ensure uninterrupted airflow. Shut the door and switch on the hot air oven.

The water molecule is the target for microwave ovens; like any other molecule with a dipole, it absorbs microwave radiation. Microwave radiation is converted into heat with high efficiency, so that "superheating" becomes possible at ambient pressure. Enormous accelerations in reaction time can be achieved, if superheating is performed in closed vessels under high pressure; a reaction that takes several hours under conventional conditions can be completed over the course of minutes

WATER BATH



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1. Check the water level and ensure it is above the heating element.
2. Connect the power supply and switch ON the water bath.
3. Set the desired temperature using the temperature controller.
4. Wait for stabilization and verify temperature with a thermometer (if needed).
5. Place samples in test tubes, flasks, or beakers inside the bath.
6. Cover the bath with a lid to minimize evaporation (if applicable).
7. After use, switch OFF, remove samples carefully.
8. Drain and clean the tank regularly to avoid contamination or scale buildup.
9. Dry and store with lid open when not in use.

COLORIMETER

A colorimeter is a device used in colorimeter that measures the absorbance of particular wavelengths of light by a specific solution. It is commonly used to determine the concentration of a known solute in a given solution by the application of the Beer–Lambert law, which states that the concentration of a solute is proportional to the absorbance. The essential parts of a colorimeter are: a light source (often an ordinary low-voltage filament lamp); an adjustable aperture; a set of colored filters; a cuvette to hold the working solution; a detector (usually a photo resistor) to measure the transmitted light; a meter to display the output from the detector. In addition, there may be: a voltage regulator, to protect the instrument from fluctuations in mains voltage; a second light path, cuvette and detector.

This enables comparison between the working solution and a "blank", consisting of pure solvent, to improve accuracy. There are many commercialized colorimeters as well as open source versions with construction documentation for education and for research. Changeable optics filters are used

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in the colorimeter to select the wavelength which the solute absorbs the most, in order to maximize accuracy to select the wavelength which the solute absorbs the most, in order to maximize accuracy. The usual wavelength range is from 400 to 700 nm. If it is necessary to operate in the ultraviolet range then some modifications to the colorimeter are needed. In modern colorimeters the filament lamp and filters may be replaced by several (light-emitting diodes) of different colors. In a manual colorimeter the cuvettes are inserted and removed by hand.

An automated colorimeter (as used in an Auto Analyzer) is fitted with a flow cell through which the solution flows continuously. The output from a colorimeter may be displayed by an analogue or digital meter and may be shown as transmittance (a linear scale from 0-100 %) or as absorbance (a logarithmic scale from zero to infinity). The useful range of the absorbance scale is from 0-2 but it is desirable to keep within the range 0-1, Because, above 1, the results become unreliable due to scattering of light. In addition, the output may be sent to a chart recorder, data logger, or computer.

DIGITAL BALANCE MACHINE

The weighing scale, normally known as Analytical Scale, or Analytical balance. The balance indicates the measured result to places. All our weighing scales have functions of unit's conversation, percent and counting parts. This balance used to take accurate weighing of compounds for various analyses, such as titrimetric, gravimetric analysis etc.

CONDUCTIVITY METER

An electrical conductivity meter (EC meter) measures the electrical conductivity in a solution. The conductance of a solution can be measured by applying an alternating electrical current to the two electrodes present in the probe. While this electrical current is applied to the solution, the cations

(+ charge) move to the negative electrode and the anions (- charge) move to the positive electrode. This movement of the ions leads to the solution to be conductive. A Conductivity meter allows us to measure the level of conductivity in solutions. Conductivity is an ability of materials (solutions, metals or gases) to pass an electric current. While all materials possess the ability to pass electric currents, the degree of such ability can vary. Substances with conductive aqueous solutions are referred to as electrolytes. These electrolytes are able to break down into ions when dissolved in water, thus creating free ions in solution. Acids, bases, and salts are examples of electrolytes. Substances with non-conductive aqueous solutions are referred to as nonelectrolytes. These substances are often composed of covalent bonds, and examples include carbon-containing compounds. Solutions with high concentration of ions will exhibit high conductivity. Solutions with low concentration of ions will result in small conductivity reading. A few factors that affect conductivity measurement are temperature, concentration of ions, and the nature of ions present in solution.

SPECTROPHOTOMETER

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1. Switch on the instrument and allow it to warm up (usually 10–15 minutes).
2. Select the desired mode (e.g., Absorbance or Transmittance).
3. Set the wavelength required for the analysis.
4. Fill a cuvette with a blank solution, wipe it clean, and place it in the sample holder. Zero or calibrate the instrument using the blank.
5. Replace the blank with the sample cuvette (filled and wiped clean).
6. Close the lid and record the absorbance or transmittance reading.
7. Repeat for all samples, cleaning the cuvette between measurements.
8. After use, remove cuvettes, turn off the instrument, and clean the sample area.
9. Document the readings and store data as per lab protocol

LABORATORY HEATING PLATE

1. Place the hot plate on a stable, heat-resistant surface in a well-ventilated area.
2. Ensure the surface of the plate is clean and dry before use.
3. Place the sample container (e.g., beaker, flask) centrally on the plate.
4. Set the desired temperature using the control knob or digital panel.
5. Switch on the hot plate and monitor the heating process.
6. Use appropriate tools (e.g., tongs, gloves) when handling hot containers.
7. Do not leave the hot plate unattended while in use.
8. After use, turn off the heating and allow the plate to cool completely.
9. Unplug the hot plate and clean the surface if necessary.
10. Store the equipment properly once cooled and cleaned

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CENTRIFUGE

- Ensure that the instrument is clean & free from dust.
- Open the upper lid by lifting it up.
- Place the centrifuge tubes in the compartments provided for it.
- The compartment is designed in such a way to place six centrifuge tubes at a time.
- Switch “ON” the mains.
- Adjust the RPM speed by the knob provided on the front panel of the instrument.
- Switch “OFF” the mains when not required

LABORATORY

GLASSWARES

BEAKER

Beakers are containers which can be used for carrying out reactions, heating solutions, and for water baths. They are for containing and transferring liquids, not for measurements.

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ERLENMAYER FLASK

Erlenmeyer flasks are conical shaped flasks, which are made of Pyrex, are safe for heating solutions, recrystallizations and chemical storage. Because of its conical shape they are best for using equipment with stopcocks such as separator funnels and burettes. They are also, like beakers, for containing and transferring liquids not for making measurements.

GRADUATED CYLINDER

Graduated cylinders are used for measuring the volumes of liquids from a few milliliters to many liters. It is important to choose the graduated cylinder according to the amount of liquid to be measured for more accurate measurements. Always read meniscus point for graduated cylinders, pipettes, volumetric flasks and burettes.

VOLUMETRIC FLASK

Volumetric flasks are used for measuring very precise and accurate amounts of a liquid and are used for such when the amount is too much for pipette or burette. They are also used for solution preparation.

PIPETTE

Pipette is a glass tube used for the delivery of a measured quantity of liquids. There are two kinds of pipettes:

- Graduated (Measuring) Pipette: has many graduated marks, much like a graduated cylinder that can deliver moderately accurate volumes (left picture)
- Volumetric (Transfer) Pipette: a kind with a bulbous middle section that has a single mark for the quantitative delivery of a single volume of liquid each Time (middle and right picture)

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	**PASTEUR PIPETTE** Pasteur pipette (or medicine dropper) is a plastic or glass pipette used to transfer small amounts of liquids, but are not graduated or calibrated for any particular volume.
	**STAND (STAND)** A metal rod attached to a heavy metal base. The heavy base keeps the stand stable, and the vertical metal rod allows for easy height adjustment of the iron ring/clamp.
	**BURETTE** Burette is a vertical cylindrical piece of laboratory glassware with a volumetric graduation on its full length and a stopcock on the bottom. It is used to dispense known amounts of a liquid reagent in a titration experiment.
	**FILTER PAPER** Filter paper is used to separate solid particles from liquids. They can have different size with different pore size. Solids that remain on filter paper can later be dried on a watch glass or in an oven liquids from one container to another. In addition, with the aid of filter paper, they can be used as separation devices to separate liquids from solids. It is fixed by a ring support on a stand
	**funnel** A separator funnel is used in liquid-liquid extractions to separate the components of a mixture between two immiscible solvent phases of different densities. It is fixed on a stand by a ring support.

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	**ROUND-BOTTOM FLASK** Round bottom flasks are used for heating or boiling of a liquid, in distillation procedures and to carry out chemical reactions. Their two or three-necked versions are also available and usually more suitable for carrying out reactions.
	**TEST TUBE** Test tubes are used as containers for solids and liquids to perform quick tests for properties such as solubility, effect of heat, etc. They can also be used as centrifuge tubes when a separation of solid and liquid is necessary
	**TEST TUBE RACK** The test tube racks provide places to hold the test tubes vertical so that chemicals are not spilled out.
	**TEST TUBE BRUSH** Test tube brush is a long and narrow equipment to clean the inside of glassware particularly test tubes.
	**THERMOMETER** A thermometer is a device used to measure temperatures or temperature changes.
	**SPATULA** Spatula or spoons are hand tools used to weigh solids in balance.

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	**STIRRING ROD** A stirring rod is made of solid glass and used to stir liquids in flasks or beakers.
	**BUNSEN BURNER** Bunsen burner is used for heating when no flammable material is present. The burner can be regulated by changing the air and gas mixture.
	**WASH BOTTLE** Wash bottles usually contain distilled water or acetone and make for a convenient method to wash out lab glasswares.
	**STOPPER** Stoppers are used to close flasks and test tubes to protect from the environment.

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List of Experiments

1. To identify the three cation radicals and three anion radicals present in the given inorganic mixture.
2. To determine the melting point of the given organic compound by the Aluminium Block Method.
3. To purify the given impure compound by sublimation.
4. To identify the functional group present in the given organic compound by elemental analysis.
5. To determine the relative viscosity of the given liquid using an Ostwald Viscometer.
6. To determine the relative surface tension of the given unknown liquid using a Stalagmometer at room temperature.
7. To determine the total hardness and permanent hardness of the given water sample by the EDTA method.
8. To identify the functional group present in the given organic compound by elemental detection and prepare suitable derivatives of the compound.
9. To determine the transition temperature of the given substance ($MnCl_2 \cdot 4H_2O$) by the thermometric method.
10. To prepare cis-Diaquabis(oxalato)ferrate(III) ion.
11. To prepare iodoform from acetone.
12. To separate the given binary mixture containing two solid compounds using water and prepare suitable derivatives of each compound.
13. To determine the concentration of the given unknown acetic acid (CH_3COOH) solution using ammonium hydroxide (NH_4OH) by conductometric titration.

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INORGANIC CHEMISTRY EXPERIMENT

CALIBRATION OF GLASSWARE AND WEIGHTS USING A CHEMICAL BALANCE

Aim

To calibrate the glassware (pipette, burette, and volumetric flask) and weights (both gram and milligram weights) using a chemical balance.

Apparatus

Pipette, burette, conical flask, volumetric flask, funnel, beaker, chemical balance, distilled water, etc.

Calibration of Glassware

In laboratory work, measuring flasks, pipettes, and burettes are used in volumetric experiments. To obtain accurate measurements of the volume of liquids delivered by these instruments, the glassware must be properly calibrated.

Pipettes

Two types of pipettes are commonly used: volumetric pipettes and graduated (measuring) pipettes. A volumetric pipette is intended for highly accurate measurements, whereas a graduated pipette is comparatively less accurate. A volumetric pipette delivers a fixed volume accurately only when used correctly.

The approximate delivery times are:

- 10 mL pipette – 20 seconds
- 25 mL pipette – 30 seconds
- 50 mL pipette – 35 seconds

If the pipette is forced to discharge the liquid too rapidly, the delivered volume may vary.

A pipette should always be held in a vertical position and allowed to drain naturally. The last drop should not be forced out by blowing or warming the bulb. Instead, the tip of the pipette should be touched gently against the inner wall of the receiving beaker or conical flask so that any freely flowing liquid can drain out. This procedure ensures that the same constant volume is delivered each time.

Calibration of Pipettes (10 mL and 20 mL)

1. Rinse the 10 mL and 20 mL pipettes with chromic acid cleaning solution and leave them immersed overnight.
2. Wash the pipettes thoroughly with tap water and finally rinse them with distilled water.
3. Fill the 10 mL pipette with distilled water up to the calibration mark.
4. Transfer the water into a previously weighed, clean, and dry conical flask.
5. Record the time required for the last drop to leave the pipette.
6. Determine the mass of the conical flask containing the water.
7. Calculate the volume of water using the related\text{Volume}=\frac{\text{Mass}}{\text{Density}}
8. Repeat the same procedure using the 20 mL pipette.

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Observations

Mass of empty, clean, and dry conical flask = M_1 = _____ g

Mass of conical flask + water = M_2 = _____ g

Mass of water = (M_2 – M_1) = _____ g

Calibration of Burette

1. Fill the burette with chromic acid cleaning solution and leave it overnight.
2. Wash the burette thoroughly with water and finally rinse it with distilled water.
3. Fill the burette with distilled water and adjust the meniscus exactly to the zero mark.
4. Deliver 5 mL of water (0–5 mL) into a previously weighed, clean, and dry conical flask.
5. Determine the mass of the conical flask containing the water and calculate the mass of the 5 mL sample.
6. Deliver the next 5 mL of water (5–10 mL) into the same conical flask, weigh it again, and calculate the mass of the additional 5 mL of water.
7. Repeat the procedure at 5 mL intervals up to 50 mL.
8. Record the density of water at room temperature.
9. Calculate the volume of water using the formula:

[\text{Volume}=\frac{\text{Mass}}{\text{Density}}]

10. Determine and record the volume correction for each interval.

Mass of 1 litre of water & volume of 1 g of water at various temperatures

Temp (°C)	Density (g/cc)	Mass	Volume of 1 g of water	Temp (°C)	Density (g/cc)	Mass	Volume of 1 g of water
20	0.9982	997.18	1.0028	24	0.9975	996.38	1.0036
21	0.9979	997.00	1.0030	25	0.9970	996.17	1.0038
22	0.9977	996.80	1.0032	26	0.9967	995.93	1.0041
23	0.9975	996.60	1.0034	27	0.9965	995.69	1.0043

Observations

- Laboratory temperature = ---° C
- Density of water at laboratory temperature =g/cc

Burette levels	Mass of water	Volume of water	Correction
0-5			
5-10			
10-15			

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15-20			
20-25			
25-30			
30-35			
35-40			
40-45			
45-50			

Calibration of Measuring Flasks (250 mL & 100 mL) Relative to a Burette

Method I (Using a Calibrated Burette)

1. Fill the **250 mL measuring flask** with chromic acid cleaning solution and leave it overnight.
2. Wash the flask thoroughly with water and dry it completely.
3. Fill the measuring flask with distilled water delivered from a **calibrated burette** until the calibration mark is reached.
4. Record the volume of water required to fill the flask up to the mark.
5. Using the mass and density of water, calculate the actual volume of the measuring flask and determine its calibration.

Method II (Using Mass of Water)

1. Fill the **100 mL measuring flask** with chromic acid cleaning solution and leave it overnight.
2. Wash the flask thoroughly with water and dry it completely.
3. Weigh the empty, clean, and dry measuring flask and record its mass.
4. Fill the flask with distilled water up to the calibration mark.
5. Remove any water droplets adhering to the outside of the flask using filter paper.
6. Weigh the measuring flask containing distilled water and record the mass.
7. Calculate the mass of the water and determine the volume using the density of water.

Observations

Mass of empty and dry measuring flask = **M_1 = _____ g**

Mass of measuring flask + water = **M_2 = _____ g**

Mass of water = **($M_2 - M_1$) = _____ g**

Laboratory temperature = **_____ °C**

Density of water at laboratory temperature = **_____ g/mL (g/cm³)**

Calibration of Weights

Weights should be calibrated to detect any inaccuracies in their actual values. This is particularly important for fractional weights supplied as separate units. All weights used with a chemical balance should be calibrated by comparing them with the corresponding standard weights from a certified weight box. Before calibration, the chemical balance should be adjusted to its zero resting point. The weights to be calibrated are then compared one by one with the corresponding standard weights.

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Gram weights are usually provided with screw tops. Any irregularity in their mass can be corrected by unscrewing the top and adjusting the quantity of fine sand or small lead shots contained in the cavity. For milligram weights, it is generally more convenient to select only those weights that are accurate and reject those showing significant variation. If required, slightly heavier milligram weights may be adjusted by gently rubbing them with emery paper or carefully trimming them with a pair of scissors.

Fractional weights	Corrected weight
500 mg	
200 mg	
200 mg	
100 mg	
50 mg	
20 mg	
20 mg	
10 mg	

Use of a Digital (Electronic) Balance

1. Place the electronic balance on a flat, stable, and vibration-free laboratory table.
2. Press the **"ON"** button and wait until the display shows **0.000 g** (or zero).
3. Always use a suitable container, such as a weighing bottle, watch glass, weighing paper, or beaker, to hold the sample. **Never place the substance directly on the balance pan.**
4. Place the empty container on the balance and press the **"TARE"** or **"ZERO"** button. The display will return to zero, indicating that the mass of the container has been automatically deducted.
5. Carefully add the required quantity of the substance to the container. If necessary, remove the container while adding the sample and then place it back on the balance.
6. Record the mass of the substance as displayed on the digital balance.
7. After use, remove the sample, clean the balance pan, and switch off the balance.

DETERMINATION OF HYDROCHLORIC ACID

Aim

To prepare a standard oxalic acid solution, standardize the sodium hydroxide solution, and determine the concentration of hydrochloric acid in the given solution.

Apparatus

Burette, pipette, conical flask, volumetric flask, funnel, beaker, measuring cylinder, etc.

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Chemicals

Oxalic acid, hydrochloric acid, sodium hydroxide, phenolphthalein indicator, distilled water, etc.

Theory

Oxalic acid dihydrate **($H_2C_2O_4 \cdot 2H_2O$; Molecular Mass = 126 g mol^{-1})**, although not a primary standard, is a satisfactory secondary standard because of its stability, non-hygroscopic nature, and good solubility in water. It is a dibasic acid having an equivalent mass of **63 g $equiv^{-1}$**.

The mass of oxalic acid required to prepare a solution of a given **normality (N)** and **volume (V)** is calculated using the equation:

$$\mathbf{W=\frac{N\times E\times V}{1000}}$$

where:

- **W** = Mass of oxalic acid (g)
- **N** = Normality
- **E** = Equivalent mass
- **V** = Volume (mL)

The standard oxalic acid solution is used to standardize sodium hydroxide, which is a secondary standard.

The sodium hydroxide solution is standardized by titration against the standard oxalic acid solution using **phenolphthalein** as the indicator. The end point is indicated by a colour change from **colourless to pale pink**.

Reaction:

$$\mathbf{2NaOH + H_2C_2O_4 \rightarrow Na_2C_2O_4 + 2H_2O}$$

The standardized sodium hydroxide solution is then used to determine the concentration of hydrochloric acid by titration using phenolphthalein as the indicator.

Reaction:

$$\mathbf{NaOH + HCl \rightarrow NaCl + H_2O}$$

Procedure

Preparation of Standard (0.05 N) Oxalic Acid Solution

1. Accurately weigh **0.7875 g** of oxalic acid.
2. Transfer it to a beaker and dissolve it in distilled water.
3. Quantitatively transfer the solution, along with the washings, into a **250 mL volumetric flask**.
4. Make up the volume to the calibration mark with distilled water.
5. Stopper the flask and shake thoroughly to obtain a homogeneous solution.

Standardization of Sodium Hydroxide Solution

1. Wash the burette with distilled water and rinse it with the given sodium hydroxide solution.
2. Fill the burette with sodium hydroxide solution up to the zero mark, ensuring that no air bubbles are present.
3. Wash the pipette with distilled water and rinse it with the standard oxalic acid solution.

4. Wash a clean conical flask with distilled water.
5. Pipette exactly **25.0 mL** of the standard oxalic acid solution into the conical flask.
6. Add **2–3 drops** of phenolphthalein indicator.
7. Titrate against the sodium hydroxide solution until the solution changes from **colourless to pale pink**.
8. Record the burette reading.
9. Repeat the titration until concordant readings are obtained and calculate the exact normality of the sodium hydroxide solution.

Determination of Hydrochloric Acid

1. Dilute the hydrochloric acid supplied in the **250 mL volumetric flask** to the calibration mark with distilled water and mix thoroughly. *(Alternatively, the sample HCl solution may be supplied directly.)*
2. Pipette **25.0 mL** of the hydrochloric acid solution into a clean conical flask.
3. Add **2–3 drops** of phenolphthalein indicator.
4. Titrate against the standardized sodium hydroxide solution until the colour changes from **colourless to pale pink**.
5. Repeat the titration to obtain concordant readings.
6. Calculate the concentration of hydrochloric acid using the appropriate formula.

Observations and Calculations

Preparation of 250 mL of Standard (0.05 N) Oxalic Acid Solution

$$\mathbf{W=\frac{N\times E\times V}{1000}}$$

$$\mathbf{=\frac{0.05\times63\times250}{1000}=0.7875\ g}$$

Mass of empty watch glass : $\mathbf{m_1}$ **= _____ g**

Mass of watch glass + oxalic acid : $\mathbf{m_2}$ **= _____ g**

Mass of oxalic acid : $\mathbf{(m_2 - m_1)}$ **= _____ g**

If the experimentally weighed mass differs from **0.7875 g**, the actual normality of the oxalic acid solution is calculated as:

$$\mathbf{\text{Normality of Oxalic Acid}=\frac{\text{Mass of Oxalic Acid}\times4}{\text{Equivalent Mass of Oxalic Acid}}}$$

or

$$\mathbf{=\frac{(m_2-m_1)\times4}{63}=N}$$

Standardization of Sodium Hydroxide

Solution in the burette : Sodium hydroxide solution

Solution in the conical flask : 25.0 mL standard oxalic acid solution

Indicator used : Phenolphthalein

End point : Colourless to pale pink

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Pink Tabulations

Trial No	Burette readings (cc)		Volume of NaOH added (cc) (B – A)	Concordant burette reading in cc (CBR)
	Initial reading(A)	Final reading(B)		
1	0.0			
2	0.0			
3	0.0			

Calculations

The following equation is used:

$$[N_1V_1 = N_2V_2]$$

Where:

$\mathbf{N_1}$ = Normality of sodium hydroxide (NaOH)

$\mathbf{V_1}$ = Volume of sodium hydroxide solution used (Concordant Burette Reading, CBR)

$\mathbf{N_2}$ = Normality of oxalic acid solution

$\mathbf{V_2}$ = Volume of oxalic acid solution taken (**25.0 mL**)

Therefore,

$$[N_1=\frac{N_2V_2}{V_1}]$$
$$[=; \boxed{;;;;;;N;;;;;;}]$$

Determination of the Amount of Hydrochloric Acid

Solution taken in the burette : Standard sodium hydroxide (NaOH) solution

Solution taken in the conical flask : 25.0 mL hydrochloric acid (HCl) solution

Indicator used : Phenolphthalein

Colour change at the end point : Colourless to pale pink

Calculations

The following equation is used:

$$[N_1V_1 = N_2V_2]$$

Where:

$\mathbf{N_1}$ = Normality of hydrochloric acid (HCl)

$\mathbf{V_1}$ = Volume of hydrochloric acid solution taken (**25.0 mL**)

$\mathbf{N_2}$ = Normality of standardized sodium hydroxide (NaOH) solution

$\mathbf{V_2}$ = Volume of sodium hydroxide solution used (Concordant Burette Reading, CBR)

Therefore,

$$[N_1=\frac{N_2V_2}{V_1}]$$
$$[=; \boxed{;;;;;;N;;;;;;}]$$

The strength of hydrochloric acid may then be calculated, if required, using:

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$$[\text{Strength (g L}^{-1}\text{)} = N \times \text{Equivalent Mass of HCl}]$$

Since the equivalent mass of HCl is **36.5 g equiv^{-1}**,

$$[\boxed{\text{Strength of HCl} = N \times 36.5\ \text{g L}^{-1}}]$$

pink Tabulations

Trial No	Burette readings (cc)		Volume of NaOH added (cc) (B –A)	Concordant burette reading in cc (CBR)
	Initial reading (A)	Final reading (B)		
1	0.0			
2	0.0			
3	0.0			

Calculations

The following equation is used:

$$[N_1V_1 = N_2V_2]$$

Where:

N_1 = Normality of hydrochloric acid (HCl)

V_1 = Volume of hydrochloric acid solution = **25.0 mL**

N_2 = Normality of sodium hydroxide (NaOH)

V_2 = Volume of sodium hydroxide used (Concordant Burette Reading, CBR)

Therefore,

$$[N_1=\frac{N_2V_2}{V_1}]$$

$$[=\boxed{;;;;;N;;;;;}]$$

We know that the **equivalent mass of hydrochloric acid (HCl) = 36.5 g equiv^{-1}**

Therefore,

Amount of HCl present in 1 dm^3 (1 L) of solution

$$[X=\text{Normality of HCl}\times\text{Equivalent Mass of HCl}]$$

$$[=N\times36.5]$$

$$[=\boxed{;;;;;g,L^{-1};;;;;}]$$

Amount of HCl present in 250 mL of solution

$$[=\frac{X}{4}]$$

$$[=\boxed{;;;;;g;;;;;}]$$

Result

S. No.	Particulars	Result
1	Normality of oxalic acid solution	_____ N
2	Normality of sodium hydroxide solution	_____ N

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3	Normality of hydrochloric acid solution	_____ N
4	Amount of hydrochloric acid present in **250 mL** of solution	_____ g

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