Lab Manual

B.Sc. (Physics, Chemistry, Mathematics)

Ist year/Ist Semester

P r e p a r e d by

Department of Chemistry
INTEGRAL UNIVERSITY

Lab Manuals
B.Sc.(PCM)Ist year/Ist semester

1) Introduction

The On-Line Lab Manual serves as your text for the lab portion of the courses B.Sc.(PCM). You must:

- Carefully read through the experiment to be performed.
- Look up information on equipment, materials and special techniques required for the experiment.
- Complete your pre-lab assignment (if given one).
- Print out hard copies of the experiment to be performed and the data sheet for the experiments.

2) Student Safety and Emergency Information:

2.1) Personal Protective Equipment (PPE) and Safe Attire

a. Wear chemical safety goggles and a knee length (41-42 inch) laboratory white coat at all times while in the laboratory when anyone is conducting experiments.
b. Wear closed shoes at all times while in the laboratory.
c. Wear nitrile gloves when directed to do so by your instructor and/or lab manual.
d. Confine long hair when in the laboratory so that it will not catch on fire or come into contact with chemicals.

2.2) Behavioral Rules for Safety

a. Do not enter the laboratory until your lab instructor is present.
b. Do not eat, drink, chew gum or smoke in the laboratory at any time. Keep all food and drinks sealed and in your backpack or purse.
c. Consider all chemicals to be hazardous unless instructed otherwise.
d. Do not taste anything in the chemistry laboratory.
e. Smell chemicals carefully and only when instructed to do so. Waft odors towards your nose rather than sniffing directly.
f. Do not use flammable liquids near open flames. Most organic liquids are flammable. Diethyl ether is especially dangerous.
g. When heating substances in a test tube, never point the mouth of the test tube at yourself or at anyone else. It may erupt like a geyser.
h. Do not force glass tubing or thermometers into rubber stoppers. The tubing or thermometer may break and cut you badly. Consult with your laboratory instructor for assistance.
i. Use caution when handling Bunsen burners, hot plates, and glassware or other equipment that has been heated. Burns are the most common laboratory injury so treat all equipment as if it were hot during experiments that involve heating.
j. Work with dangerous or volatile chemicals in a fume hood as directed by your instructor and/or lab manual.
k. Do not perform unauthorized experiments. If you see someone else doing something you think may be dangerous, tell him or her to stop and/or report the incident to your lab instructor. If another student tells you to stop doing something because it is unsafe, stop as directed. Consult your lab instructor if there is a problem or difference of opinion.

2.3) Handling Accidents

a. Notify your lab instructor immediately if you have an accident, spill, or are injured in any way.
b. If chemicals come in contact with your skin or eyes, wash with water for at least 15 minutes. 18. Know where to find and how to use the eyewash stations in the lab. It is not recommended to wear contact lenses in the laboratory since chemicals splashed in the eye may get under the lens therefore be difficult to rinse. If a splash occurs while you are wearing contact lenses, they must be safely removed as quickly as possible.
c. Know where to find and how to use the safety shower in the front of the room.
d. Clean up spilled chemicals immediately. Consult your laboratory instructor if you are not sure what to do.
e. Solid sodium bicarbonate (baking soda) is available in the laboratories in containers located by the sinks. Use this to neutralize acid spills before wiping them up. Similarly, solid citric acid solution is available in containers by the sinks and should be used to neutralize base spills before wiping them up. A saturated solution of sodium bicarbonate is also available by the sinks and can be used to wipe dried acid or base residue off of lab benches as needed. However, if acid or base spills on your skin, don't waste time looking for these neutralizing substances. Rinse with water immediately for at least 15 minutes.

2.4) Proper Waste Disposal

Separate waste as follows:

a. Waste chemicals should be disposed of as directed by your lab instructor. Most chemicals are NOT to be thrown down the sink. Special waste receptacles will be provided for these chemicals. Waste chemicals must be sorted by kind, not just mixed with other, different waste chemicals. Read waste container labels carefully. Notify your instructor when a waste bottle is nearly full. Do not overfill waste bottles.
b. Broken glass is to be disposed of in the cardboard boxes labeled "Broken Glass Only" located near the doors to the lab. A dustpan and broom are located in each lab to assist you in cleaning up broken glass. Do not put broken glass in the regular trash, and do not put anything except broken glass in the broken glass containers!
c. Gloves used in lab are to be disposed of in the containers labeled “Used Gloves Only” located next to the sinks in each lab.
d. Other trash that is not glass and is not contaminated by hazardous chemicals should be placed in the large waste baskets near the front of the lab room.

2.5) Other Information You Should Know

a. Material Safety Data Sheets (MSDS) are available for all the chemicals used in this course. These sheets give information about the chemical, physical, and physiological properties of chemical substances. See your instructor for information about accessing these sheets. A shortcut to MSDS websites is available on the site mention in the table of contents. They can also be found by entering the name of the chemical and MSDS into Google or any other search engine.
b. Each laboratory experiment involves its own specific hazards. Be sure to read your laboratory procedure carefully before arriving for lab, and take note of all safety precautions. You are

responsible for the information provided in the laboratory procedure. You must also arrive on time for all laboratory sessions so you will be present to hear the safety information provided by your lab instructor. For the safety of all students in the class, students who arrive late to lab will not be allowed to perform the lab experiment that day.

2.6) Student Safety Training Record

Department Chemistry Laboratory Student Safety Training Record

Course:_____Semester:_____ Year:_____

Instructor:_____Date of Training:_____

I certify that I have read the online available following documents from the Chemistry Department, and that I agree to abide by the policies therein:

1. Chemistry Laboratory Safety Rules

2. Emergency Procedures for chemistry lab Classes

3. Instructions for the Safe Use and Care of Chemistry Laboratory Goggles, Coats & Gloves.

S.No.	Enrol. No.	Name of Student	Course name	Year/Semester	Signature	Do you wear contact lenses under your goggles? This information may be needed in case of an emergency.
						___yes ___no
						___yes ___no
						___yes ___no
						___yes ___no

3) Chemical Hygiene Plan (CHP)

I. Purpose

This Chemical Hygiene Plan (CHP) sets forth policies, procedures, equipment, personal protective equipment and work practices that are capable of protecting employees and students from the health hazards presented by hazardous chemicals used in laboratories. This Plan is intended to meet the requirements of Occupational Exposure to Hazardous Chemicals in Laboratories

II. Scope

This plan applies to our Chemistry Laboratory where employees work with substances in containers that are easily and safely manipulated by one person. The objective of this program is to provide guidance to all laboratory personnel who use chemicals, so that they can perform their work safely.

Laboratory Employees -- Each individual working in a laboratory should be informed about

hazards associated with that laboratory and the specific work going on there. This includes all faculty, laboratory staff and student workers.

Support Personnel -- Storeroom, janitorial, maintenance, and delivery personnel may be exposed to potential physical and chemical hazards from work carried out in the laboratory. They must be informed about the risks involved and trained how to avoid potential hazards.

Department Head, Faculty members, Lab instructors, Lab attendants shall:

1. Work with administrators, faculty and laboratory staff to develop and implement appropriate chemical hygiene policies and practices;
2. Monitor procurement and use of chemicals in the lab, determining that laboratory facilities and training levels are adequate for chemicals in use;
3. Perform regular, formal chemical hygiene and housekeeping inspections that include inspections of emergency equipment;
4. Maintain a current chemical inventory of chemicals present within the lab and store room;
5. Review and improve the Chemical Hygiene Plan on, at a minimum, an annual basis.
6. Maintain overall responsibility for the safe operation of the laboratories.
7. Determine the proper level of personal protective equipment; ensure that such protective equipment is available and in working order;
   Ensure that the appropriate training has been provided to employees;
8. Monitor the waste disposal program.

III. Standard Operating Procedures for Laboratory Chemicals

A. Chemical Procurement

The decision to procure a new chemical shall be made by the appropriate Department Head who will ensure a commitment to safe handling and use of the chemical from initial receipt to ultimate disposal.

Department of Chemistry is continually and aggressively evaluate current inventory and properly dispose of unnecessary materials.

Requests for procurement of new chemicals (i.e. those not currently included in a department‟s chemical inventory – this does not apply to re-orders of substances already in use) shall be submitted to the appropriate Department Head for approval.

A requisition form shall be used for this purpose. Chemicals used in the laboratory shall be those that are appropriate for the ventilation system. All chemicals must be received in the chemistry storage room. Personnel who receive chemicals shipments shall be knowledgeable of the proper procedures for receipt.

Chemical containers shall not be accepted without accompanying labels, material safety data sheets (MSDS). All chemical shipments should be dated when received and opened.

B. Chemical Storage

The storage area shall be well illuminated, with storage maintained at or below eye level.

Flammables will be stowed in the designated flammable storage cabinets in lab prep area**s**.

Chemicals must be segregated by hazard classification and compatibility in a well-identified area, with good general exhaust ventilation.

Mineral acids should be segregated from flammable and combustible materials. Acid resistant trays shall be placed under bottles of mineral acids. Nitric acid will be stored in an acid cabinet. Acid sensitive materials, such as cyanides and sulfides, shall be separated from acids and protected from contact with acids and water. Highly toxic chemicals or other chemicals whose containers have been compromised shall be stored in unbreakable secondary containers. The storage area shall NOT be used as a preparation or repackaging area. The storage area shall be accessible during normal working hours.

Stored chemicals shall be examined at least annually by the Lab instructors for container integrity and/or deterioration. The inspection should determine whether any corrosion, deterioration, or damage has occurred to the storage facility as a result of leaking chemicals.

The Lab instructors shall conduct periodic inventories of chemicals outside the storage area.

Unneeded items shall be properly discarded or returned to the storage area.

C. SUGGESTED CHEMICAL STORAGE PATTERN:

The common method of storing the chemicals in alphabetical order sometimes results in incompatible shelved materials. For example, storing strong oxidizing materials next to organic chemicals can present a hazard.

A possible solution is to separate chemicals into their organic and inorganic families and then to further divide the materials into related and compatible families. Below is a list of compatible families.

INORGANIC

1. Metals, Hydrides
2. Acetates, Halides, Iodides, Sulfates, Sulfites, Halogens, Thiosulfates, Phosphates
3. Amides, Nitrates (except Ammonium Nitrate), Nitrites, Azides
4. Hydroxides, Oxides, Silicates, Carbonates
5. Sulfides, Selenides, Phosphides, Carbides, Nitrides
6. Bromates, Perchlorates, Perchloric Acid, Chlorites, Hypochlorites, Peroxides, Hydrogen Peroxide
7. Arsenates, Cyanides, Cyanates
8. Borates, Chromates, Manganates, Permanganates
9. Acids (except Nitric). Store acids in a designated cabinet. *Nitric Acid is isolated and stored by itself.
10. Sulfur, Phosphorus, Arsenic, Phosphorus Pentoxide

ORGANIC

1. Acids, Anhydrides, Peracids
2. Alcohols, Glycols, Amines, Amides, Imines, Imides
3. Hydrocarbons, Esters, Aldehydes
4. Esters, Ketones, Ketenes, Halogenated Hydrocarbons, Ethylene Oxide
5. Epoxy Compounds, Isocyanates
6. Sulfides, Polysulfides, Sulfoxides, Nitriles
7. Phenols, Cresols

ADDITIONAL STORAGE SUGGESTIONS

1. No floor chemical storage.
2. No top shelf chemical storage.
3. No reactive liquid chemicals stored above eye level.
4. Shelf assemblies are firmly secured to walls. Avoid island shelf assemblies.
5. Provide anti-roll-off lips on all shelves.
6. Ideally shelving assemblies would be of wood construction.
7. Avoid metal, adjustable shelf supports and clips. Better to use fixed, wooden supports.
8. Store acids in dedicated acid cabinet(s). Store nitric acid in that same cabinet ONLY if isolated from other acids. Store both inorganic and some organic acids in the acid cabinet.
9. Store flammables in a dedicated and ventilated flammables cabinet.
10. Store severe poisons in a dedicated poisons cabinet.
11. Segregate known or suspect carcinogens from other chemicals.
12. If you store volatile materials (ether, hydrocarbons, etc. in a refrigerator, the refrigerator must be explosion-proof. The thermostat switch or light switch in a standard refrigerator may spark and ignite volatile vapors in the refrigerator.)

D. Chemical Handling

Each laboratory employee (with training, education, and resources provided by supervision) shall develop work habits consistent with requirements of the Department of Chemistry CHP to minimize personal and coworker potential exposure to chemicals. Based on the realization that all chemicals inherently present hazards in certain conditions, exposure to all chemicals shall be minimized.

General precautions that shall be followed for the handling and use of all chemicals are:

1. The amount of chemicals at the lab bench shall be as small as practical.
2. Skin contact with hazardous chemicals shall be avoided at all times.
3. Employees shall wash all areas of exposed skin prior to leaving the laboratory. Soap is provided at each sink.
4. Mouth suction is prohibited for pipetting or starting a siphon.
5. Eating, drinking, smoking, chewing gum, or application of cosmetics in the laboratories prohibited.
6. Storage of food or beverages is not allowed in storage areas or refrigerators used for laboratory operations.
7. All chemicals and equipment shall be properly labeled, in accordance with Department of Chemistry CHP guidelines.
8. Any chemical mixture shall be assumed to be as toxic as its most toxic component.
9. Substances of unknown toxicity shall be assumed to be toxic.
10. Laboratory employees shall be familiar with the symptoms of exposure for the chemicals that they work with and the precautions necessary to prevent exposure.
11. All laboratory employees shall adhere to the CHP.
12. Specific precautions based on the toxicological characteristics of individual chemicals shall be implemented as deemed necessary by the CHP.

E. Laboratory Equipment and Glassware

Each employee shall keep the work area clean and organized. At the completion of each workday or operation, the work area shall be thoroughly cleaned and all equipment cleaned and stowed. In addition, the following procedures shall apply to the use of laboratory equipment:

a. All laboratory equipment shall be used only for its intended purpose.
b. All glassware will be handled and stored with care to minimize breakage; all broken glassware will be immediately disposed of in the broken glass container.
c. All evacuated glass apparatus shall be shielded to contain chemicals and glass fragments should implosion occur. Heavy-walled filtration flasks connected to aspirators or house vacuum lines are excepted.
d. Labels shall be attached to all chemical containers, identifying the contents and related hazards.
e. Waste receptacles shall be clearly labeled.
f. All laboratory equipment shall be inspected on a periodic basis and replaced or repaired as necessary.
g. Engineering controls and safety equipment in the laboratory shall be utilized and inspected in accordance with guidelines established in the CHP.
h. The appropriate Laboratory Technician shall maintain an inspection log that documents monthly eyewash/shower testing and flushing. A sticker indicating the date of last flushing shall be placed on each shower or eyewash station.
i. The appropriate Laboratory Technician shall visually inspect fire extinguishers monthly. A log of the date of the last visual inspection shall be posted by each extinguisher. Regular maintenance of fire extinguishers is the responsibility of SMC‟s Facilities Department.

F. Personal Protective Equipment

a. Safety goggles are required for employees and visitors to the Chemistry laboratories and will be worn at all times when chemicals are being used in the laboratory.
b. The wearing of contact lenses in the laboratory is strongly discouraged.
c. Chemical goggles and/or a full-face shield shall be worn during chemical transfer and handling operations as procedures dictate.
d. Lab coats should be worn in the laboratory.
e. Appropriate chemical-resistant gloves shall be worn at all times when there exists the potential for skin contact with hazardous chemicals.
f. Used or contaminated gloves are to be disposed of in the special glove disposal containers in each lab. Contaminated gloves must not be worn outside of the laboratory. Thermal resistant gloves shall be worn for operations involving the handling of heated materials and exothermic reaction vessels.

G. Personal Work Practices

2. Department Head must ensure that each employee knows and follows laboratory-specific rules and procedures established by this plan. Faculty must ensure that enrolled students receive

appropriate instruction in laboratory safety polices.

2. All employees shall remain vigilant to unsafe practices and conditions in the laboratory and shall immediately report such practices and/or conditions to the Department Head. The Head must PROMPTLY correct unsafe practices or conditions.
3. Long hair or loose-fitting clothing shall be confined close to the body to avoid contact with chemicals or being caught in moving machine/equipment parts.
4. Avoid unnecessary exposure to hazardous chemicals by any route. Do not smell or taste any laboratory chemicals.
5. Encourage safe work practices in coworkers by setting the proper example. Horseplay is strictly forbidden.
6. Seek information and advice from knowledgeable persons regarding Standards and Codes about hazards present in the laboratory and plan operations, equipment, and protective measures accordingly.
7. Use engineering controls (fume hoods, safety shields and general ventilation) in accordance with CHP procedures.

H. Labeling

1. All containers in the laboratory shall be labeled. This includes chemical containers and waste containers. The labels shall be informative and durable, and at a minimum, will identify contents, source, date of acquisition, and indication of hazard.
2. Portable containers shall be labeled by the individual using the container. Exemptions for labeling requirements shall be made for chemical transfers from a labeled container into a container that is intended only for the immediate use of the employee who performed the transfer.

IV. Criteria for Implementation of Control Measures

A. When to use fume hoods:

Hoods should be used WHENEVER POSSIBLE to contain and exhaust toxic, offensive, or flammable materials. Processes that have potential for generating hazardous airborne chemical concentrations must be carried out within a fume hood.

B. When to use personal protective equipment:

Eye Protection - Safety goggles must be worn by all personnel in the laboratory whenever hazardous chemicals are in use. NO EXCEPTIONS.

Gloves - Gloves should be worn to protect the skin from chemical and physical (e.g. heat, cold) exposures. Used or contaminated gloves are to be disposed of in the special glove disposal containers in each lab. Contaminated gloves must not be worn outside of the laboratory. Thermal resistant gloves shall be worn for operations involving the handling of heated materials and exothermic reaction vessels. Thermal resistant gloves shall be non-asbestos and shall be replaced when damaged

or deteriorated.

Laboratory Coats – Knee-length white laboratory coats are to be worn by all employees and students while working with laboratory chemicals.

V. When to institute special work practices:

The Department Head must approve special work practices. If particularly hazardous chemicals are to be used (e.g. carcinogens, reproductive toxins, teratogens, or acutely toxic chemicals), standard operating procedures for the use of these substances must be developed and followed.

VI. Fume Hood Management

A. Frequency and type of monitoring - all local exhaust hoods used for primary containment control will be monitored for adequate airflow annually. The survey will be completed with a calibrated velometer.

B. Acceptable operating range - Minimum face velocities of at least 100 linear fpm must be maintained for each hood.

C. Maintenance schedule - Maintenance of local exhausts or fume hoods will be completed on an "as needed" basis, or annually, whichever comes first.

VII. Employee Information and Training

Employees will be provided with training to ensure that they are apprised of the hazards of chemicals present in their work area. Such training will be provided at the time of an employee's initial assignment to a work area where hazardous chemicals are present and prior to assignments involving new exposure situations.

VIII. Procedures to secure medical consultation and examination are as follows:

a. Seek immediate medical care at IIMSR.
b. Report exposure to instructor, faculty member or Department Head.
c. The following information will be provided to the physician.
d. Identity of hazardous chemical.
e. Description of conditions under which exposure occurred.
f. Description of signs and symptoms employee is experiencing
g. Copy of MSDS.
h. A written opinion from the physician shall be provided to the employer including:
    i. Recommendation for further medical follow-up.
    j. Results of medical exam and tests.
    k. Any medical condition revealed during the exam that places the employee at increased risk.
    l. A statement that the employee has been informed by the physician of the results of the exam and any medical condition that may require further treatment or examination.

IX. Emergency Response/Chemical Spills

a. When spills of hazardous chemical occur within the Laboratory, the following procedures are followed to prevent injury or property loss:

b. Provide any first aid (if necessary) to affected individuals. Liberally use eyewash station and/or safety shower to **flush affected areas for AT LEAST 15 minutes**. A large exposure to the body merits ambulatory service.

c. Notify HOD of spill.
d. Evacuate the area.
e. Always refer to MSDS for special precautions or spill cleanup requirements.
f. If spilled materials exhibit flammability, eliminate ignition sources such as hot plates, Bunsen burners, etc., if this can be done safely.
g. Avoid all contact with spilled material. If necessary, use protective gloves, gown, goggles, and/or respirator.
h. Neutralize acids and bases.
i. Contain collected materials and label container with name of contents and also as Hazardous Waste.

Liquid Spills

a. Confine spill to as small an area as practical.
b. For small quantities of acids or bases, use the neutralizing agent from the chemical spill clean-up kit. An absorbent material specially prepared for acid/base spills may also be used.
c. For small quantities of other materials, such as organic solvents, utilize an absorbent material to clean-up spill. Examples of absorbent materials are vermiculite, dry sand, paper towels, etc.
d. For large quantities of inorganic acids and bases, flush with large amounts of water, preferably toward a containment area. *CAUTION must be taken not to add too much water to create a flood that may react with water-reactive materials and cause spattering and additional personnel exposure.
e. If possible, with small manageable spills, utilize spilled containment material (kitty litter, sand, or booms) found in the emergency spill kits located throughout the Science Departments. Large quantity spills will be handled by professional hazardous waste personnel or the fire department.
f. Carefully pick up and decontaminate any bottles, broken glass, and/or other containers. Decontaminate over the bucket or pail to collect contaminated wash.
g. Avoid using any shop vacuum that is not rated for chemical clean up. A potential exists for atomizing hazardous wastes and creating a potential human inhalation exposure.
h. If the spill is extremely volatile (high vapor pressure), allow the spill to evaporate and exhaust out the laboratory exhaust (e.g., fume hood).
i. Properly contain, label, store and/or dispose of collected hazardous waste. (See waste disposal section for methods).

Solid Spills

Sweep solid spill of low toxicity into a designated, easily decontaminated, dust pan and place in a labeled container for disposal.

Additional Spills

Mercury - Clean up with a mercury spill clean-up kit. Collect elemental mercury in a sealed container

to prevent exposure to mercury vapors. In the event of large spills or spills that render some mercury unavailable for clean up (e.g. mercury in floor cracks or beneath lab benches), an airborne evaluation of mercury vapor content may be required.

Compressed Gas Cylinders

Any compressed gas cylinders used in science laboratories must be secured with two chains, top and bottom, at all times when in use and stored. In addition, all cylinders must be properly labeled. Regulators must not be left attached to unused cylinders for extended periods of time.

Incident Report

An incident investigation should take place after each spill and/or accident. The Incident Report should be completed by concerned instructor and faculty member and forwarded to the HOD.

XII. Review and Update

This Chemical Hygiene Plan will be reviewed and updated annually.

Instructions for the Safe Use and Care of
Chemistry Laboratory Coats, Goggles & Gloves

Chemical Splash Goggles:

1. Purchase a pair of chemical safety goggles).
2. Bring your goggles with you for all laboratory sessions of your chemistry class. You will not be allowed to work in the lab without your goggles.
3. Wear your goggles when anyone in the lab is conducting an experiment.

1. Purchase a lab coat that fits you well. Lab coats that are too tight or too loose are not safe. Sleeves that are too long should be rolled up.
2. If your lab coat has not been contaminated with a hazardous substance, you may wash it as you do your other clothing.
3. If your lab coat becomes contaminated with a hazardous substance, as with any other lab spill, notify your instructor immediately.
4. Contaminated lab coats will be handled by your instructor as they deem appropriate.

Nitrile Gloves:

a) Nitrile gloves are to be worn only during portions of experiments where specified by the experimental procedure, when instructed by the instructor or supervisor, or when working with substances for which the protocol requires the use of gloves.
b) Note that nitrile gloves are flammable and will stick to your skin if they burn. Do not wear gloves while working with Bunsen burners.
c) Do not wear gloves outside the lab.
d) When a chemical comes in contact with a glove, remove the glove immediately and place it in the glove waste.
e) Do not touch surfaces such as door knobs, computer keyboards, and chairs while wearing

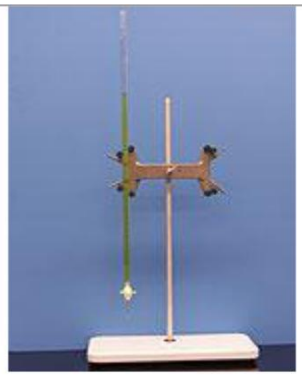
gloves.

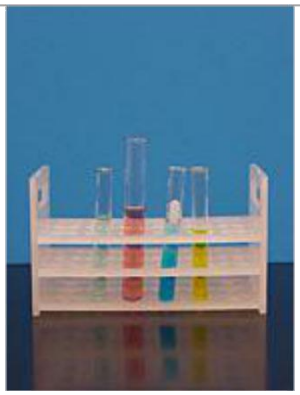
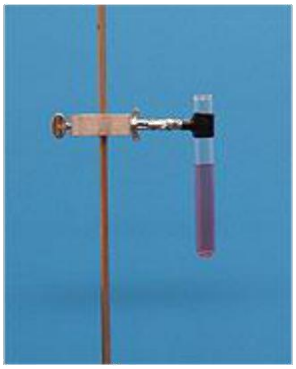
f) Gloves with holes or tears must be removed immediately and disposed of properly.
g) Dispose of gloves at the end of each experiment in the glove waste containers provided in each lab.

4) MSDS Sheets Online: http://hazard.com/msds/

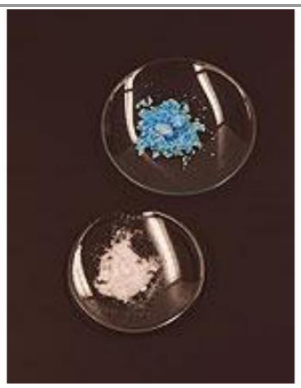
5) Chemistry Laboratory Common Instruments:

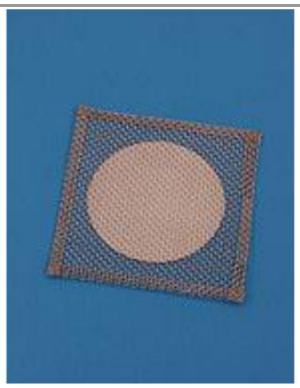
Below are photos and names of common lab equipment you will encounter in Chemistry lab listed in alphabetical order.

 Balance (electronic)	 Beakers	 Bunsen Burner	 Buret
 Clay Triangle	 Crucible	 Crucible in Triangle	 Crucible Tongs
 Dropper Pipets	 Dropper in action	 Erlenmeyer Flasks	 Evaporating Dish

 Forceps	 Funnels	 Goggles	 Graduated Cylinders
 Pinch Clamp	 Pipets and Bulbs	 Plastic and Rubber Policemen	 Ring Clamp & Stand
 Scoopula	 Stirring Rods	 Thermometers	 Test Tubes in Rack
 Test Tube Holder	 Tube & Holder in Action	 Utility Clamp	 Clamp in action


Wash Bottle


Watch Glasses


Wire Gauze


Combined for Heating

An additional site to view lab equipment, including techniques for using it, may be found at: http://www.dartmouth.edu/~chemlab/techniques/ph.html

6) Required Materials:

Following materials are required to perform the experiments in the chemistry lab.

- **Safety Goggles:** Chemical splash goggles are required for all laboratory experiments. Safety goggles must fit snugly to your face, and be able to fit over your prescription eye wear.
- **Laboratory Coat:** A knee length (41-42 inch) laboratory white coat must be worn at all times while in the laboratory when anyone is conducting experiments.
- **Closed Shoes:** Wear closed shoes at all times while in the laboratory.
- **Nitrile Gloves:** Nitrile gloves must be worn when directed to do so by your instructor and/or by the lab manual.
- **Scientific Calculator:** This calculator should preferably be equipped with log, ln, exp and 1/x functions.
- **Lab Notebook:** Purchase one note book for recording the experiments that you will perform.

7) Instruction for Lab record writing:

1. **Write on the right hand page the following order:**

a. Serial number and date of performance (in the margin)
b. Name and number of the experiment as given in the list.
c. Aim of the experiment.
d. Description of the apparatus.
e. Procedure including sources of error and precautions taken to eliminate or to minimize them.
f. Inference or Result.
g. Explanation, if necessary of any divergence in the expected result.

2. **Left hand page should contain the following in their proper places**.

a. Neat diagram of the main apparatus.
b. Observation in tabular form.
c. Calculation in tabular form.
d. Graph sheets and other papers to be attached.

3. Students should submit a record of the previous experiments when they come for practical work.

4. An experiment is deemed to be complete when it is satisfactorily performed and recorded.

8) Basic Concepts of Volumetric Analysis

Chemical analysis of the compounds is carried out in two ways

1. Qualitative analysis.
2. Quantitave analysis.

Qualitative analysis shows what element a given contains. Quantities analysis determines the quantity of a particular component present in substance. It is carried out in two ways

1. Gravimetric analysis.
2. Volumetric analysis.

Gravimetric analysis involves the estimation of the amount of a given compound from the results of weighing. Volumetric analysis is based on the measuring the volume of the solution of a substance.

Terms involved in volumetric analysis:

1. Titration: The process of finding out the volume of one of the solution required to react completely with a definite volume of one the other solution of known concentration is called titration.

2. Titrant: The solution of known strength is called titrant.

3. Titrate: The solution whose concentration to be estimated.

4. Indicator: The reagent which indicates the endpoint or equivalent point of the titration. The strength of concentration of a solution is expressed in the following ways.

NORMALITY: Number of gram equivalents of the substance dissolved per liter of the solution is called Normality. It is denoted by N Normality = Wsolute/Esolute × 1/Vsovent (in lit) Where E is Gram equivalent weight

MOLARITY: Number of grams moles of a solute dissolved per liter of solution is called Molarity. It is denoted by M

Molarity = Wsolute/Msolute × 1/Vsovent (in lit)

Where M is Gram molecular weight

MOLALITY: It is the number of mole of the substance dissolved in 1kg of the solvent it is denoted by (m).

Molality = Wsolute/Msolute × 1/Wsovent (in kg)

9) List of Experiments:

(For B.Sc. PCM 1st Year /1st Sem)

Subject Name: Chemistry Practical-I, Subject Code: CH118

1. Preparation of standard solution related to normality & molarity.
2. Preparation of buffer solution, pH measurement.
3. Acid – base titration.
4. Oxidation-reduction (redox) titration.
    a. To determine the strength of oxalic acid.
    b. To determine the strength of ferrous ammonium sulphate (Mohr's salt) solution by using external indicator.
5. To determine the strength of potassium permanganate solution by using sodium thiosulphate solution Iodometrically.
6. To determine the strength of given copper sulphate solution by using thiosulphate solution Iodometrically.
7. Complexometric titration.
    a)To estimate the concentration of calcium ions with EDTA.
    b) To estimate the concentration of magnesium ions with EDTA.
8. Detection of element present in the given organic compounds.
9. Detection of function group present in the given organic compounds.

    a. Carboxylic
    a. Phenolic
    b. Alcoholic
    c. Aldehydic
    d. Ketonic
    e. Ester
    f. Amine
    g. Amide.

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(To be pasted on the first page of Lab Note Book)

Subject Name: Industrial Chemistry Lab-I **Subject Code: CH1111**

Name of Student: **Course:** **Group:**

S.No.	EXPERIMENT	DATE	SIGNATURE	GRADE
1.	Preparation of standard solution related to normality & molarity.			
2.	To Prepare buffer solutions of different pH.			
3.	To prepare approximately 0.1 N NaOH & standardize it with 0.1N oxalic acid.			
4.	To determine the strength of Ferrous Ammonium Sulphate (Mohr's salt) solution by using external indicator.			
5.	To determine the strength of potassium permanganate solution by using sodium thiosulphate solution Iodometrically			
6.	To determine the strength of given copper sulphate solution by using thiosulphate solution Iodometrically.			
7.	To estimate the concentration of Ca^{2+} & Mg^{2+} ions with EDTA.			
8.	Detection of element present in the given organic compounds			
9	To Detect the presence of function groups in the given organic compound.			

Grade Average:

Signature of Teacher:

Name of Teacher:

EXPERIMENT No. 1

Object: Preparation of standard solutions related to normality and molarity.

1- Standard Hydrochloric Acid Solution (0.1 N).
2- Standard Sodium thiosulphate Solution (0.1 N).
3- Standard Sulphuric Acid solution (0.1N)

Introduction: A solution of known normality is called a standard solution. Its concentration is determined by a process known as standardization. If we have a primary standard (a compound which is very pure, stable, non-hydroscopic, and with a high molecular weight), we can prepare a standard solution simply by dissolving a known amount of the compound in a known volume of liquid. If we don't have such a compound, we shall have to standardize our solution against a primary standard.

Standard Hydrochloric Acid Solution (0.1 N):

Reagents: Anhydrous sodium carbonate- primary standard.
Methyl orange indicator

Glassware: Burette, Pipette, beaker ,conical flask,funnel etc.

Procedure:- Transfer by means of graduated cylinder or a burette 44.5 ml or 49.1 ml of concentrated hydrochloric acid (HCl) into a 1000 ml volumetric flask containing about 500 ml of water.Cool and dilute to 1000 ml.Standardize the solution as follows.

Take 0.3 g of anhydrous sodium carbonate into 250 ml conical flask,dilute to 40 ml with water,and add 3 drops of methyl orange.Titrate the sodium carbonate solution against the hydrochloric acid solution to be standardize ,continue the addition the colour due methyl orange becomes a very faint yellow.Boil the solution and cool again.Continue the titration by adding the acid drop-wise and carefully until the colour of methyl orange becomes orange or faint pink.The titration is repeated with two other portions of sodium carbonate solution. Calculate the normality of hydrochloric acid as follows.

Calculation:

$$\text{Normality of HCl} = \frac{\text{Wt.of } Na_2CO_3 \times 1000 \times \% \text{ Purity}}{\text{Titre Volume (ml)} \times 53.0 \times 100}$$

Standard Sodium Thiosulphate Solution (0.1 N):

Reagents:
Potassium dichromate
Potassium iodide
Conc.HCl
Sodium thiosulphate
Starch solution (0.5%):Mix 0.5 g of soluble starch with about 15 ml of water and pour into 100 ml of hot water. Boil for 102 min.

Glassware: Conical flask, burette, pipette.

Procedure:

Weigh 25 g of sodium thiosulphate crystals ($Na_2S_2O_3.5H_2O$),dissolve in water and dilute 1000 ml. Standardize the solution as follows:

Weigh accurately 0.125 g of ground potassium dichromate into a 250 ml conical flask and 25 ml of distilled water.Add 2 g of potassium iodide and 8 ml of concentration hydrochloric acid.Mix thoroughly and titrate with sodium thiosulphate, swirling the liquid constantly until the brown colour changes to yellowish green.Add 2 ml of starch solution and continue the titration until the colour changes sharply from blue to light green.Repeat the titration two more times.Calculate the normality of sodium thiosulphate solution on the basis of normality of potassium dichromate.

Calculation:

$$\text{Normality of } Na_2S_2O_3 \text{ solution} = \frac{\text{g of } K_2Cr_2O_7 \times 1000 \times \%\ \text{Purity}}{\text{Titre Volume (ml)} \times 49.03 \times 100}$$

Standard Sulphuric Acid Solution (0.1 N):

Reagents: Standard NaOH solution (0.1 N).
Phenolphthalein indicator solution.

Glassware: Conical flask,volumetric flask,burette,pipette.

Procedure: Transfer 3 ml of sulphuric acid into a 1000 ml volumetric containing about 500 ml of water.When cold,dilute to 1000 ml.Standardize the solution as follows:

Transfer 25 ml of standard 0.1 N sodium hydroxide solution into 250 ml conical flask.Titrate with sulphuric acid solution using phenolphthalein indicator.Repeat the titration with two more 25 ml portion of sodium hydroxide solution.The various titration should agree within 0.1 ml.Calculate the normality of sulphuric acid solution on the basis of standard sodium hydroxide solution.

Calculation:

$$\text{Normality of } H_2SO_4 = \frac{\text{Normality of NaOH} \times 25}{\text{Titre Volume (ml)}}$$

Viva-Voce Questions:

1. What is normality? Write down its formula.
2. What is morality?
3. What do you mean by standard solution?
4. What do you mean by neutralization?
5. What is volumetric analysis?

EXPERIMENT No. 2(a)

Object: Preparation of acetate buffer solution (pH range 3- 6).

Chemicals required: 1) 0.1 M acetic acid
2) 0.1 M sodium acetate (tri-hydrate) (13.6gm/L)

Glassware: Beaker, measuring cylinder, pipette funnel etc.

Procedure: Mix the solution in the following proportion to get the required pH.

S.No.	pH	Volume of 0.1 M acetic acid	Volume of 0.1 M sodium acetate
1.	3	982.3 ml	17.7 ml
2	4	847.0 ml	153.0 ml
3	5	357.0 ml	643.0 ml
4	6	52.2 ml	947.8 ml

Result: Check the pH of prepared solution with pH meter and report the values.

Viva-Voce Questions:

1. What do you mean by pH of a solution?
2. What is the effect of temperature on pH?
3. Who suggested the term pH?
4. What is buffer solution?

EXPERIMENT No. 2(b)

Object: Preparation of buffer solution of pH 7 and 4.

Chemicals required: (1) 0.1 M NaOH (2) 0.1M potassium dihydrogen phosphate

(3) Sodium dihydrogen phosphate (4) disodium hydrogen phosphate

(5) 0.1 M potassium hydrogen phthalate (6) disodium hydrogen phosphate

(7) Potassium hydrogen phosphate

Glassware: Beaker, measuring cylinder, pipette funnel etc.

Procedure:

(A) For pH = 7.00

Add 29.1 ml of 0.1 molar NaOH to 50 ml 0.1 molar potassium dihydrogen phosphate.

OR Alternatively,

Dissolve 1.20 g of sodium dihydrogen phosphate and 0.885 g of disodium hydrogen phosphate in 1 liter volume distilled water

(B) For pH = 4.00

Add 0.1 ml of 0.1 molar NaOH to 50 ml of 0.1 molar potassium hydrogen phthalate.

OR Alternatively,

Dissolve 8.954 g disodium hydrogen phosphate $12H_2O$ and 3.4023g of potassium Hydrogen phosphate in 1 liter volume distilled water.

Result: Check the pH of prepared solution with pH meter and report the values.

1.
2.
3.
4.

Viva-Voce Questions:

1. What is buffer solution?
2. What do you mean by pH? Draw the pH scale.
3. Why is necessary to know the pH of solution?
4. What is the electrochemical cell?

EXPERIMENT No. 2(c)

Object: Preparation of phosphate buffer.

Principle: Phosphate salts are known by serval names and the correct phosphate must be used to prepare buffer solution.
One phosphate cannot be substituted for another phosphate. Check formula of salt to certain.

Chemical Formula	Name of salt	Other name
KH_2PO_4	Potassium dihydrogen phosphate.	Potassium dihydrogen orthophosphate.
K_2HPO_4	Potassium hydrogen phosphate.	Dipotassium hydrogen orthophosphate.
K_3PO_4	Potassium phosphate	Tribasic potassium phosphate

Chemicals required:

1) 0.1 M disodium hydrogen phosphate (14.2g/l)
2) 0.1 M HCl
3) 0.1 M NaOH

Glassware: Beaker, measuring cylinder, pipette funnel etc.

Procedure: Mix the solutions in the following proportions to get the required pH solution.

pH	Volume of phosphate	Volume of 0.1 M HCl	Volume of 0.1 M NaOH
7	756.0 ml	244 ml	
8	955.1 ml	44.9 ml	
9	955.0 ml	45.0 ml	
10	966.4 ml		

Result: Check the pH of prepared solution with pH meter and report the values.

1.
2.
3.
4.

Viva-Voce Questions:

1. What is difference between calomel and glass electrode?
2. How the equivalence point in acid base titrations is determined pH metrically?
3. Why pH meter should be calibrated before used?
4. How can you prepare 0.1 M NaOH solution?

EXPERIMENT No. 3(a)

Object: To prepare 250 ml of approximately 0.1N Hydrochloric acid (HCl) and standardize it with 0.1N sodium carbonate (Na_2CO_3).

Theory: Substance which are not available in pure state e.g. mineral acids and caustic alkalis are prepared as approximate solutions and standardized against known pure substance solution (primary standard). It is a direct titration method which is carried out by adding the solution of titrant (acid) from the burette to the solution of base (Na_2CO_3) containing a suitable indicator.

Chemical equation:

$Na_2CO_3 + 2HCl \longrightarrow 2NaCl + CO_2 + H_2O$

Chemicals: Concentrated Hydrochloric acid (HCl), Sodium Carbonate (Na_2CO_3)

Apparatus: Volumetric flask (250 ml), conical flask, beaker, burette, pipette, funnel.

Indicator: methyl red (pH 4.2 - 6.3)

End point: yellow to pink

Preparation of 0.1 N HCl :

Equivalent weight of HCl = 36.5

1000 ml of 1N HCl = 36.5 gm of HCl

1000 ml of 0.1 N HCl = 3.65 gm of HCl

As commercially available conc. HCl is 36%--38% w/w i.e.

36 gm of HCl = 100 gm of conc. HCl

1 gm of HCl = 100/36

3.65 gm of HCl = 10.1 gm of conc. HCl

Density of HCl = 1.18 gm /L

Volume = mass/density

= 10.1 / 1.18

= 8.5 ml of conc. HCL

For 1000 ml of 0.1 N HCl:

8.5 ml of commercial available conc. HCl is required to be diluted with Distilled water to 1000 ml.

For 250 ml of 0.1 N HCl:

= 8.5/4 = 2.1 ml commercial available conc. HCl. So 2.1 ml conc. HCl is required to be diluted to 250 ml with distilled water.

Preparation of 0.1 N Sodium Carbonate (Na_2CO_3)

Molecular weight of Na_2CO_3 = 106

Equivalent weight of Na_2CO_3 = 106 / 2

= 53.00

1000 ml of 1N Na_2CO_3 = 53.00 gm of Na_2CO_3

1000 ml of 0.1N Na_2CO_3 = 5.30 gm of Na_2CO_3

250 ml of 0.1N Na_2CO_3 = 1.32 gm of Na_2CO_3

Calculations:

(HCl) (Na_2CO_3

$N_1V_1 = N_2V_2$

Procedure: Weigh accurately 1.32 gm of anhydrous sodium carbonate (Na_2CO_3).Dissolve it in 250 ml of distilled water. Pipette out 25 ml of 0.1N Na_2CO_3 solution in a conical flask and add 2 or 3 drops of methyl red indicator. Add HCl solution from the burette until the colour of solution changes from yellow to faint pink. Repeat the titration to get concordant readings

Observation Table:

S.No.	**Volume of sodium carbonate (Na_2CO_3) solution taken(ml)**	**Burette reading**		**Concordant Volume of HCl solution used (ml)**
		Initial	**Final**	
1				
2				
3				

Result: The strength of given HCl solution is _____

Viva-Voce Questions:

1. What is standardization?
2. How can you prepare 0.1N HCL solution?
3. What is the colour of methyl red/ orange in acidic and basic medium?
4. Find equivalent weight of Na_2CO_3?
5. What are the commercial uses of Na_2CO_3?

EXPERIMENT No. 3(b)

Object: To prepare 250 ml of approximately 0.1N sodium hydroxide (NaOH) and standardize it with 0.1N oxalic acid.

Theory: An acid–base titration is the determination of the concentration of an acid or base by exactly neutralizing the acid or base with an acid or base of known concentration. This allows for quantitative analysis of the concentration of an unknown acid or base solution. It makes use of the neutralization reaction that occurs between acids and bases and the knowledge of how acids and bases will react if their formulas are known. Acid–base titrations can also be used to find percent purity of chemicals. The reaction between oxalic acid and sodium hydroxide is given below.

Chemical equation:

$H_2C_2O_4$ (aq) + 2 NaOH (aq) == $Na_2C_2O_4$ (aq) + 2 H_2O (l)

Chemicals: sodium hydroxide (NaOH), oxalic acid $(COOH)_2 .2H_2O$

Apparatus: Volumetric flask (250 ml), conical flask, beaker, burette, pipette, funnel.

Indicator: Phenolphthalein (pH 8.3 - 10)

End point: pink to colourless

Procedure: Fill the burette with 0.1N oxalic acid solution. Pipette out 25 ml of NaOH solution in a conical flask and 2 or 3 drops of phenolphthalein indicator. The colour of solution becomes pink. Now add drops oxalic acid solution from the burette till the colour of the solution changes from pink to colourless. Repeat the titration to get concordant readings.

Observation Table:

S.No.	Volume of Sodium Hydroxide (NaOH) solution taken (ml)	Burette reading		Concordant volume of oxalic acid solution used (ml)
		Initial	Final	
1				
2				
3				

Calculations:

Preparation of 0.1 N sodium hydroxide (NaOH)

Molecular weight of NaOH = 40

Equivalent weight of NaOH = 40

For 250 ml: Weigh accurately 1 gm of NaOH and dissolve in distilled water.

Preparation of 0.1N oxalic acid

Molecular weight of oxalic acid = 126

Equivalent weight of oxalic acid = 63

For 250 ml: Weigh accurately 1.575 gm of oxalic acid and dissolve in distilled water

(NaOH) (Oxalic acid)

$N_1V_1 = N_2V_2$

Result: The strength of given NaOH solution is _____

Viva Voce questions:

1. How can you prepare 0.1N oxalic solution?
2. What is the colour of phenolphthalein in acidic and basic medium?
3. Write name of indicator are used is this experiment?

EXPERIMENT No. 4(a)

Object: To determine the strength of ferrous ammonium sulphate (Mohr's salt) solution by using external indicator

Theory: Potassium dichromate ($K_2Cr_2O_7$) oxidizes ferrous ion (Fe^{2+}) present in ferrous ammonium sulphate into ferric ion (Fe^{3+}) in acidic medium (in presence H_2SO_4) .The end point can be noted by using potassium ferricyanide $K_3[Fe(CN)_6]$ as an external indicator. Potassium ferricyanide gives a greenish blue color with ferrous ion due to the formation of ferrous ferricyanide ($Fe_3[Fe(CN)_6]_2$)

Reactions :

$$Cr_2O_7^{2--} + 14H^{+} + 6e^{--} \longrightarrow 2\,Cr^{3+} + 7\,H_2O$$

$$[\,Fe^{2+} \longrightarrow Fe^{3+} + e^{--}\,] \times 6$$

$$Cr_2O_7^{2--} + 14H^{+} + 6Fe^{2+} \longrightarrow 2\,Cr^{3+} + 7\,H_2O + 6\,Fe^{3+}$$

$$3\,Fe^{2+} + 2\,[Fe(CN)_6]^{3--} \longrightarrow Fe_3[Fe(CN)_6]_2$$

ferrous ferricyanide
(blue colour)

Chemicals: N/20 Potassium dichromate ($K_2Cr_2O_7$), ferrous ammonium sulphate ($FeSO_4.(NH_4)_2SO_4.6\,H_2O$), dilute H_2SO_4

Indicator: Potassium ferricyanide $K_3[Fe(CN)_6]$

Glassware: Burette, Pipette, Beaker, Conical flask, funnel, glass rod, measuring cylinder

Materials: White glazed tile

End point: Strong blue colour changes to light yellow colour.

Procedure:

1. Pipette out 25 ml of sample solution into conical flask and add 10 ml dilute H_2SO_4.

2. Put few drops of Potassium Ferricyanide Indicator on white glazed tile.

3. Titrate the solution against N/20 Potassium Dichromate solution.

4. Now take drop of solution from conical flask, put it on the potassium ferricyanide drop on white tile. If blue colour appears, it indicates that all ferrous ions are not converted to ferric ions.

5. Again titrate with $K_2Cr_2O_7$ solution till the drop from conical flask solution turns drop of indicator to light yellow colour. This indicates the end point.

6. Note the volume of $K_2Cr_2O_7$ from burette and repeat procedure till concordant readings are obtained .

Observation Table:

S.No.	Volume of ferrous ammonium sulphate solution taken (ml)	Burette reading		Concordant Volume of N/20 Potassium dichromate ($K_2Cr_2O_7$) solution used (ml)
		Initial	Final	
	Magnesium	**Sodium**		**Potassium**
1				
2				
3				

Calculations :

$$N_1 \times V_1 = N_2 \times V_2$$
$$N_1 \times 25 = N_2 \times V_2$$
$$N_1 = \frac{1}{20} \times \frac{V_2}{25}$$

Strength of given ferrous ammonium sulphate = Normality x Equivalent weight

$= N_1 \times 392$

$= X$ gms/lit.

392 gms of ferrous ammonium sulphate contains = 56 gm Fe

X ,, ,, ,, ,, ,, $= \frac{56}{392} \times X$

= --------gm

Result: The Iron content in the given sample solution is ___ gms/lt.

Viva Voce questions:

1. What is Mohr's salt? Write dwon its chemical formula.
2. Name of external indicator which is used in $K_2Cr_2O_7$ titration?
3. What will happen if external indicator $K_3[Fe(CN)_6]$ is used as an internal indicator.
4. What is redox titration?
5. Why sulphuric acid solution added in the preparation of Mohr's salt solution?
6. What is the IUPAC name of $K_3[Fe(CN)_6]$.

EXPERIMENT No. 4(b)

Object: Determination of the strength of given unknown oxalic acid solution by titrating it against Potassium permanganate.

Theory: Potassium permanganate is a powerful oxidizing agent and is employed for the estimation of reducing substances like oxalic acid, ferrous sulphate etc.
In acidic medium (dilute H_2SO_4) oxalic acidic oxidized by $KMnO_4$ into CO_2 and H_2O.

$$2\ KMnO_4 + 3H_2SO_4 \text{ ---------- } K_2SO_4 + 2MnSO_4 + 3H_2O + 5O$$
$$5\ H_2C_2O_4 + 5O \text{ --------- } 10CO_2 + 5H_2O$$

$$2KMnO_4 + 3H_2SO_4 + 5H_2C_2O_4 \text{ -------- } K_2SO_4 + MnSO_4 + 8\ H_2O + 10CO_2$$

The above reaction proceeds slowly at room temperature but at 60 – 70 °C quantitative oxidation takes place. As soon as the oxidation of oxalic acid is complete, addition of a drop of $KMnO_4$ produces a permanent pink colour in the solution, indicating the end point. In this titration no need of indicator as $KMnO_4$ itself acts as an indicator.
Chemicals: 0.1 N Potassium permanganate ($KMnO_4$), 0.1N oxalic acid ($H_2C_2O_4$), dilute H_2SO_4
Apparatus: Volumetric flask (250 ml), conical flask, beaker, burette, pipette, funnel
Indicator : Potassium permanganate (self indicator)
End point: colourless to pink
Procedure: Rinse and fill the burette with $KMnO_4$ solution. Pipette out 25 ml of oxalic acid solution in a clean conical flask and add to it 10 ml dilute H_2SO_4 . Heat the contents to about 60 – 70 °C . Then run in $KMnO_4$ solution from the burette, quickly in the beginning with constant stirring . The colour disappears. Continue adding $KMnO_4$ solution dropwise, till a permanent light pink colour persists for about 30 seconds. Repeat the titrations till two concordant readings are obtained.

Observation Table:

S.No.	Volume of oxalic acid solution taken(ml)	Burette reading		Concordant Volume of $KMnO_4$ solution used (ml)
		Initial	Final	
1				
2				
3				

Calculations:

$$N_1V_1 = N_2V_2$$

Result: The strength of given oxalic acid solution is _____

Viva Voce Questions:

1. How many types of indicator? Which type of indicator is used in this experiment.
2. What do you mean by strength? Give the formula of strength.
3. How can you prepare 0.1 N Potassium permanganate in 500 ml water?
4. Write name of chemical used as oxidizing agent? And why it is used.

EXPERIMENT No. 5

Object: To determine the strength of given potassium permanganate solution by titrating it against sodium thiosulphate solution iodometrically.

Chemicals: N/20 potassium permanganate solution,N/20 sodium thiosulphate,potassium iodide, H_2SO_4.

Glassware: Pipette, burette, conical flask, beaker etc.

Indicator: Starch solution

End point: Blue to colourless.

Theory: In acedic medium potassium permanganate oxidizes potassium iodide and iodine is librated.The liberated iodine is then titrated against sodium thiosulphate using starch as an indicator.

$$2KMnO_4 + 8H_2SO_4 + 10KI \rightarrow 6K_2SO_4 + 2MnSO_4 + 6H_2O + 5I_2$$

$$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI.$$

Procedure: Fill the burette with sodium thiosulphate solution.Pipette out 25 ml potassium permanganate solution in 250 ml conical flask.Add to the conical flask 10-15 ml of dil.H_2SO_4 followed by 5 ml of 25% KI solution.Cover the flask with watch glass and keep it in a dark place for 2-3 minutes.The solution turns brownish in colour due to liberation of iodine.Run the sodium thiosulphate solution from the burette into conical flask with constant shaking .Continue the addition of thiosulphate solution dropwise with constant shaking till the solution in the conical flask becomes light or pale yellow colour. Add 1-2 ml of starch solution to this yellow coloured solution.The solution in conical flask immediately turns blue.Continue the dropwise addition of sodium thiosulphate solution until the blue colour just disappears.This is the end point.Note the volume of sodium thiosulphate used.Repeat the titration to get more readings

.

Observation:

S.No.	Volume of the potassium permanganate solution taken(ml)	Burette reading		Volume of sodium thiosulphate used (ml)
		Initial	Final	
1				
2				
3				

Calculation:

$$N_1V_1 = N_2V_2$$

$$N_1 = \frac{N_2 V_2}{V_1}$$

Strength of potassium permanganate = Eq.wt. x Normality

Result : The strength of unknown $KMnO_4$ solution is…….gm/litre

Viva Voce questions:

1. What do you mean by iodometric titration?
2. What is hypo solution? Write down the name & formula.
3. Write the name of indicator used in this experiment.
4. What is the difference between iodimetery and iodometery?
5. Why always excess of KI is used?
6. What is the advantage of using starch as indicator in iodine titration?

EXPERIMENT No. 6

Object: To prepare a standard N/20 copper sulphate solution and then determine the strength of sodium thiosulphate solution iodometrically.

Theory: When copper sulphate reacts with potassium iodide, cupric iodide is formed. This is unstable and soon liberates iodine

$$2CuSO_4 + 4KI \longrightarrow 2CuI_2 + 2K_2SO_4$$
$$2CuI_2 \text{ ---------- } 2\,CuI + I_2$$

---

or $2CuSO_4 + 4KI$ ---------- $2CuI + I_2 + 2K_2SO_4$

$KI + I_2$ ---------- KI_3

The iodine so liberated remains dissolved in excess of KI and is proportional to the amount of copper sulphate which is then titrated against sodium thiosulphate using starch as indicator. At the end point the blue colour disappears and a white ppt. of cuprous iodide is obtained.

$$Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2\,NaI$$

sodium tetra thionate

Chemicals: N/20 $CuSO_4.5H_2O$, N/20 sodium thiosulphate ($Na_2S_2O_3.5H_2O$) ,solid KI.
Glassware: Burette, Pipette, Beaker, conical flask, funnel, measuring cylinder, etc.
Indicator: Freshly prepared starch solution.
End point: Disappearance of Blue colour.

Procedure: Rinse and fill the burette with sodium thiosulphate (hypo) solution. Pipette out 25 ml of copper sulphate solution in a clean conical flask and add 1 gm of KI (or 1 ml of 100 % KI solution). The colour of the solution becomes brown. Run in sodium thiosulphate (hypo) solution from burette with constant shaking, till the colour of the solution becomes light yellow. Add about 2 ml of starch solution. A blue colour is developed .Continue addition of sodium thiosulphate (hypo) solution drop by drop till blue colour just disappears. This is the end point. At this stage the colour of the ppt. will be white due to the formation of CuI. Repeat the titrations to get two concordant values

Precautions: Sometimes the white ppt. of cuprous iodide (CuI) becomes dirty brown due the adsorption of iodine by it. This may lead to difficulty in determine the end point. This can be avoided by adding 5 ml of 10 % potassium or ammonium thiocynate to the titration mixture just before adding the indicator.

Observation Table:

S.No.	Volume of copper sulphate solution taken (ml)	Burette reading		Concordant Volume of sodium thiosulphate (hypo) solution used (ml)
		Initial	Final	
1				
2				
3				

Calculations:

$N_1V_1 = N_2V_2$

Strength of sodium thiosulphate solution = Normality x equivalent weight

Result: The strength of given sodium thiosulphate solution is _____

Viva Voce questions:

1. Why the solution mixture kept in the dark?
2. Why starch gives blue colour with iodine in presences of iodide?
3. Why starch solution added near the end point of an iodine titration and not in the beginning?
4. Why only freshly prepared starch solution should be used?

EXPERIMENT No. 7(a)

Object: Determination of concentration of Ca^{2+} ions by EDTA using Eriochrome Black –T as indicator.

Chemicals: Calcium carbonate, Buffer solution, 0.01M EDTA, dil.HCl.

Glassware: Pipette, Burette, conical flask, beaker etc

Indicator: Eriochrome Black-T

End Point: Red to blue colour.

Procedure: Pipette out 25 ml of freshly prepared calcium carbonate solution in 250 ml conical flask. Dilute this solution by adding 25 ml of distilled water. Add 2 ml of buffer solution and 5-6 drops of indicator.Titrate this solution with 0.01M EDTA solution filled in the burette until the colour of the solution changes from red to blue. Repeat the titration in order to get at least two more readings.

Observation and Calculations:

S.No	Volume of calcium carbonate solution taken	Volume of EDTA solution used
1.		
2.		
3.		

Calculate the amount of Ca^{2+} from the following relationship

1 ml of 0.01 M EDTA = 0.4008 mg Ca

OR

1 ml of 0.1M EDTA = 4.008 mg Ca

Result: The strength of calcium in the given calcium solution =

$[V_1 \times 0.4008/1000] \times 100$ %

Note: Ca^{2+} solution (0.01M):- Weigh accurately 1.0 gm of calcium carbonate (Eq.wt 100.09) and transfer it in a 1 litre measuring flask containing distilled water. Add dil.HCl drop by drop till there is effervescence and the salt completely dissolves. Now make the solution upto 1 litre mark by adding distilled water.

EDTA solution: For 0.01M solution dissolve 0.93 gm of disodium salt of EDTA in 250 ml of redistilled water.

EXPERIMENT No. 7(b)

Object: Determination of concentration of Mg^{2+} ions by EDTA using Eriochrome Black as indicator.

Chemicals: Ethylenediamine tetraacetic acid (H_4Y), Eriochrome Black-T indicator, Buffer solution (pH-10), 0.01M Magnesium sulphate.

Glassware: Pipette,Burette,Conical flask Beaker etc.

Indicator: Eriochrome Black-T.

End Point: Red to blue colour

Procedure: Pipette out 25 ml freshly prepared magnesium sulphate solution in a 250 ml thoroughly cleaned conical flask.Dilute this solution upto 100 ml by adding distilled water.Add 5 ml of buffer solution and 5-6 drops of indicator.Titrate this solution with EDTA solution filled in the burette until the colour of the solution changes from red to blue. Repeat the titration in order to get at least two concordant readings.

Observation and Calculations:-

S.No.	Volume of magnesium sulphate solution used.	Volume of EDTA solution used	Mean value
1.			
2.			
3.			

1 ml of 0.1 M EDTA = 2.432 gms of Mg

1 ml of 0.01 M EDTA = 0.2432 gms of Mg

V_1 ml of EDTA = 0.2432 x V_1 gms of Mg

V_1 ml of 0.01 M EDTA = 25 ml of .01 M $MgSO_4.7H_2O$.

Since 25 ml of 0.01 M $MgSO_4.7H_2O$ = 2.49/40 = 0.0624 gm of $MgSO_4.7H_2O$

0.0624 gm of $MgSO_4.7H_2O$ contain V_1 x 0.2432 gm of Mg

1 gm of $MgSO_4.7H_2O$ contains V_1 x 0.2432/1000 gm of Mg

100 gm of $MgSO_4.7H_2O$ contains [V_1 x 0.2432/1000] x100 gm of Mg

Result: The strength of magnesium in the given magnesium solution = [V_1 x 0.2432/100] x 100 %

Viva Voce questions:

1. What do you mean by complexometric titration?
2. Why EDTA is widely used for detection of metal ions?
3. What is the indicator used in EDTA titrations?

4. Give the applications of EDTA titration?

EXPERIMENT No. 8

Object : To detect the presence of Elements in the given organic compound .

Chemicals : Ferrous sulphate solution , Ferric chloride solution solution , sodium nitroprusside solution Lead acetate solution , Dilute acetic acid Dilute H_2SO_4 ,Sodium metal

Glassware : Beaker , Test tube, ignition tube, funnel ,etc .

Materials : Test tube holder, Test tube stand, Test tube brush ,Tongs ,etc

Procedure :

Lassainge's Test or Preparation of sodium Extract : Take small piece of sodium metal , dry it between the folds of filter paper to remove kerosene oil and place it into the ignition tube .Put a little amount of the given organic compound .Hold the ignition tube with the help of pair of tongs and heat gently in the flame, keeping mouth of the tube away from your face Heat the ignition tube till red hot. Plunge the red hot tube into 100 ml beaker containing 15 ml distilled water. Repeat the process with at least three more ignition tube. Boil the beaker for 10 minutes and then filter it. This filtrate is known as sodium fusion extract. It should always be clear and colourless. It contains N, S , and halogens in water soluble ionic form and is used for the detection of these elements. The extract should be alkaline. If it is not alkaline do it by adding 1 or 2 drops of NaOH.

Observation table :

S.No	EXPERIMENT	OBSERVATION	INFERENCE
1	Test for Nitrogen Take 2 ml of sodium extract in a clean test tube and add few drops of freshly prepared ferrous sulphate solution. A dirty green ppt will be obtained; in case it is not obtained , add 2 or 3 drops of NaOH solution . Now warm the solution and add few drops of dilute H_2SO_4 or dilute HCl	a prussian blue(ink colour) colour or ppt	Nitrogen present
2	Test for sulphur (i)To 2 ml of of sodium extract add few drops of freshly prepared sodium nitroprusside solution. (ii) To 2 ml of of sodium extract add few drops of acetic acid and lead acetate solution.	violet colour a black ppt.	Sulphur present Sulphur present
3	Test for Nitrogen and sulphur Take 1 ml of sodium extract in a clean test tube and add few drops ferric chloride solution and HCl	blood-red colour .	Both Nitrogen and sulphur present
4	Test for halogens Acidify 2 ml of sodium extract with dilute HNO_3 and add $AgNO_3$ solution	Observe the property of the precipitate as below : (i)*White precipitate* , soluble in NH_4OH and reappears on adding dilute HNO_3	chlorine present

		(ii) *Light yellow precipitate* dissolves in NH_4OH with difficulty and reappears on adding dilute HNO_3	bromine present
		(iii)*Yellow precipitate* insoluble in NH_4OH	iodine present

Reactions :

When nitrogen is present

$2Na + 2C + N_2$ -------- 2 NaCN
sodium cyanide

When sulphur is present

$2Na + S$ -------- Na_2S

When nitrogen and sulphur both present together

$Na + C + N + S$ --------- NaCNS
sodium sulphocyanide

When halogens are present

$2Na + Cl_2$ ------ 2NaCl
$2Na + Br_2$ ------- 2NaBr
$2Na + I_2$ ------- 2NaI

Result : The following elements are present in the given organic compounds _____

Viva Voce questions:

1. What is Lassaigne's extract?
2. Why is the sodium metal placed in kerosene oil?
3. What is the purpose of fussing the organic compound with sodium metal for preparing this solution?
4. The Lassaigne's extract of an organic compound gives a blood red colour with ferrous sulphate.what do you infer?

EXPERIMENT No. 9

Object: To detect the presence of functional groups in given organic compound.
Chemicals : Sodium bi-carbonate solution , ferric chloride solution , dilute HCl ,sodium Hydroxide solution , cerric ammonium nitrate solution , sodium nitrite solution , sodium nitroprusside solution , Schiff's reagent ,
Glassware: Beaker, Test tube, funnels, etc.
Material: Test tube holder Test tube stand Test tube brush, Tongs, etc

OBSERVATION TABLE

S.No.	Experiment	Observation	Inference
1	Carboxylic acids test (i)*Litmus Test:* Place a trace of the substance on a moist blue litmus paper *(ii) Sodium bicarbonate test:* To 5 ml of dilute solution of $NaHCO_3$ add a pinch of organic compound.	Blue litmus turns to red. A brisk effervescence due to evolution of CO_2.	May be Carboxylic (-COOH) group. Carboxylic (-COOH) group present.
2	Phenols test (i) *Litmus Test:* Place a trace of the substance on a moist blue litmus paper (ii) *Ferric chloride test*: To 2 ml of aqueous or alcoholic solution of the substance add 1 drop of $FeCl_3$ solution. (iii) *Liebermann's test*: Take a small quantity of phenol + minute crystal of sodium nitrite + 2 ml of conc. H_2SO_4 in a dry test tube and warm. An intense green or blue colour appears. Dilute with water ----- red solution; add excess of NaOH solution.	Blue litmus turns to red. A violet, blue, green, red, or reddish-brown color appears. Blue or bluish-green colour.	May be phenolic (Ar-OH) group Phenolic (Ar-OH) group Phenolic (Ar-OH) group
3	Alcohols (i) To 1ml of the substance add few drops of cerric ammonium nitrate solution. (ii) Take 1 ml of the substance in a dry test tube add a small piece of sodium.	A red colour is produced A brisk effervescence due to evolution of H_2 taken place.	Alcoholic (- OH) group is present Alcoholic (- OH) group is present.

4	Aldehydes (i) To 2 ml of 2:4 dinitrophenyl hydrazine reagent add few drops of alcoholic solution of the given substance and shake. (ii*) Schiff's reagent test* : To 2 ml of Schiff's reagent add few drops of the given substance shake vigorously and wait for 1-2 minutes (iii) *Crismer's test:* To 2 ml of Nessler's reagent add a little of the substance (iv) *Tollen's reagent test*: To 2 ml of reagent add 2 or 3 drops of the substance and heat on a water bath for about 5 minutes.	A yellow–orange precipitate formed A pink or purple colour appears A yellowish-brown, red-brown or black ppt A greyish black ppt. or silver mirror colour appears	May be aldehydic (-CHO) group aldehydic (-CHO) group present aldehydic(-CHO) group present aldehydic (-CHO) group present
5	Ketones (i) To 2 ml of 2:4 dinitrophenyl hydrazine reagent add few drops of alcoholic solution of the given substance and shake. (ii) *Sodium nitroprusside test* : To 2 ml of aqueous solution of original substance add 2 ml of sodium nitroprusside solution and make it alkaline by adding few drops of NaOH solution	A yellow–orange precipitate formed. A wine red colour changing to yellow on standing.	May be Ketonic group (C=O) Ketonic group(-C=O) present
6	Carbohydrates *Molisch test:* To 2 ml of aqueous solution of the substance add 2 or 3 drops of 10% solution of alcoholic α-naphthol and shake.Then add about 2 ml of conc. H_2SO_4 carefully along the sides of test tube.	A red-violet ring is formed at the junction of two layers .On shaking the whole solution becomes violet red with a blue ppt.	Carbohydrate present.
	Esters (i)Phenolpthalein test: To 1 ml of the substance add 2 ml water and a drop of NaOH solution and phenolphthalein.	A pink colour appears. Place the test tube in boiling water for 5 to 10 minutes. Pink colour disappears.	Ester present

7	(ii) Hydroxamic test: To a little amount of the substance add 1 ml of hydroxylamine hydrochloride dissolved in methyl alcohol and make it alkaline with methanolic KOH. Boil and cool. Acidify with dilute HCl and add few drops of $FeCl_3$.	A reddish violet colour appears.	Ester present
8	<u>Amino group</u> Take small amount of given organic substance in a clean test tube and add 2 ml of dilute HCl followed by 2 ml of sodium nitrite solution and shake gently.	Yellow oily layer	$-NH_2$

Viva Voce Questions:

1. What do you mean by functional group?
2. If compound turns blue litmus to red, what will you guess?
3. How can you detect the presence of alcoholic groups.
4. What is the difference between alcoholic & phenolic groups?
5. How can you detect the presence of aldehydes & ketones.

General precautions during experiments:

1. The apparatus should be cleaned before the start of experiment.
2. The same amount of indicator should be used in all titrations.
3. The reaction mixture should be shaken properly during titrations.
4. Titration is to be carried out at room temperature.
5. The end point of the titration should be noted carefully.
6. All reagents should be used freshly prepared.
7. Starch indicator always used should be freshly prepared.
8. Always place lid on the buffer solution bottle immediately after use.
9. Preparation of acid solutions must be carried out in ice-bath and handle carefully.
10. The pH meter should be standardized first by using a buffer of known pH.
11. The electrode must be washed properly and dried before dipping in the acid solution.
12. The temperature control knob of the pH meter should be adjusted to the room temperature.
13. Sodium metal must be kept in kerosene oil to avoid inflammation.

Precautions during titration:

1. Usually an air bubble is present in the nozzle of the burette; it must be removed before taking the initial reading.
2. There should not be any leakage from the burette during titration.
3. Keep your eye in level with the liquid surface while taking the burette reading or while reading the pipette or measuring flask etc.
4. Always read lower meniscus in case of colourless solution and upper meniscus in case of coloured solutions.
5. Do not blow through the pipette to expel the last drop of solution from it simply touch the inner surface of the titration flask with the nozzle of the pipette for this purpose.
6. Shaking of the titration flask should be continuous during adding the solution from the burette.
7. Use your index finger while pipetting the solution.

General Viva-Voce Questions:

Q. What do you mean by volumetric analysis?

Ans. Volumetric analysis is quantitative analysis involving measurement of the volumes of the solution.

Q. What do you mean by a standard solution?

Ans. A solution whose strength or concentration is known is called a standard solution.

Q. What do you mean by normality?

Ans. The number of gram-equivalent weight of substance dissolved in one litre of the solution is called as normality. It is denoted by "N".

Mathematically represented as:

$$\text{Normality} = \frac{\text{Strength in gm. /litre}}{\text{Equivalent weight of dissolved substance}}$$

Q. What do you mean morality?

Ans. Molarity is the number moles (gram molecular mass) of the solute present per litre of the solution.

$$\text{Morality} = \frac{\text{Gram of solute/litre of solution}}{\text{Gram molecular mass of the solute}}$$

\Q. What do you mean by titration?

Ans. Involves the process of finding out of the volume of the titrant required to react completely with a known volume of the solution under analysis is known as Titration.

Q. What do you mean by „End Point" in titration?

Ans. End point means completion of the reaction between the two solutions.

Q. What is meant by the terms titrant and titre?

Ans. The solution taken in the titration flask is called titrant and the solution which is made to react with it is called titre.

Q. What is indicator? Give the types of indicator.

Ans. A substance which shows a visible change in colour at the end point is called an indicator. Indicator is of three types.

a) **Internal indicator**: An indicator which is added to the reaction mixture to indicate the end point of titration. Example-- Phenolphthalein & Methyl orange etc,
b) **External indicator:** An indicator which is not added to reaction mixture. But it is used externally to indicate end point of the titration. Example: Pot. Ferricyanide
c) **Self indicator:** When one of the reactants itself acts as indicator and no external substance is required to indicate the end point of titration. Example: $KMnO_4$.

Q. Name the types of titrations?

Ans. According to the volumetric determinations can be divided into the Following methods.

a) Acid-base titration or Neutralization titrations: Neutralization titration involves the titration of a base with an acid.
b) Oxidation-Reduction titrations (redox titration): The reactions which involve simultaneous oxidation and reduction are called redox reactions and the titrations involving redox reactions are called redox titrations.
c) Precipitation titrations: Precipitation titration results in the formation of a precipitate. An example for this type is the titration of silver nitrate against sodium chloride.
d) Complexometric titrations: In complexometric titration, a complexin reagent forms complex ions with metal ions like Ca^{2+} and Mg^{2+} Sodium salt EDTA is use as a complexing reagent in titrations.

Q. What is normality equation?

Ans. The normality equation is

$$N_1 V_1 = N_2 V_2$$

List of Instruments:

S.No.	Name of Instrument	Make	Model	Quantity	Year of Purchase
1	Double Distillation Assembly	Perfit	Model 202/02	01	2014-15
2	Electronic Balance	Wensar	HPB 2000	01	2013-14
3	Hot Air Oven	Ambassador	Digital	01	2011-12
.4	Visible Spectrophotometer	Labtronics	LT- 38	01	2011-12
5	pH meter digital	Labtronics	LT-24	01	2011-12
6	Magnetic stirrer with Hot plate	Science tech	____	01	2011-12
7	Heating mantle	Science tech	____	03	2011-12
8	Water bath	Science tech	____	02	2011-12
9	Refrigerator	L.G.	____	01	2011-12
10	Electronic Balance	Sartorious	____	01	2006