Lab Manual

B.Sc. (Physics, Chemistry, Mathematics)

I^{st} year/ II^{nd} semester

P r e p a r e d by

Department of Chemistry
INTEGRAL UNIVERSITY

B.Sc.(PCM) Chemistry Lab Manuals

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

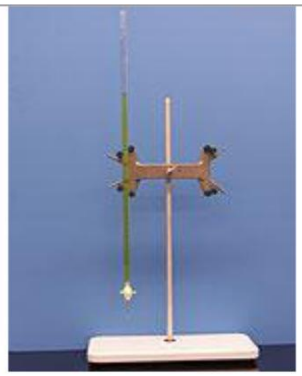
gloves.

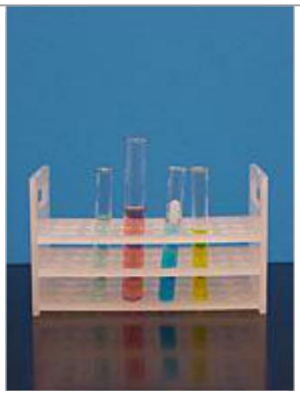
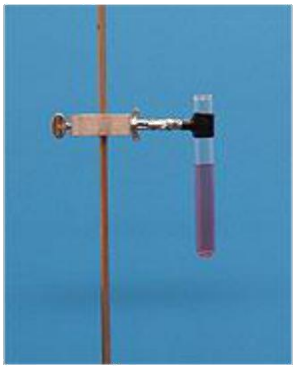
f) Gloves with holes or tears must be removed immediately and disposed of properly.
g) Dispose of gloves at the end of each experiment in the glove waste containers provided in each lab.

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

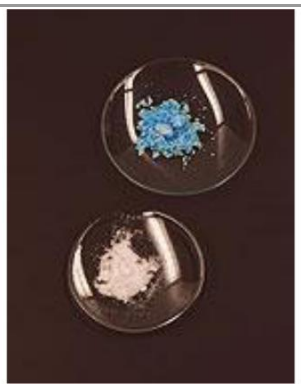
5) Chemistry Laboratory Common Instruments:

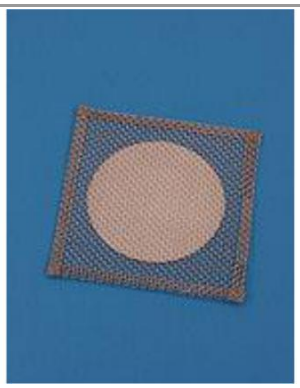
Below are photos and names of common lab equipment you will encounter in Chemistry lab listed in alphabetical order.

 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 /IInd Sem)

Subject Name: Chemistry Practical -II, Subject Code: CH120

1. To determine chloride content in the given water sample.
2. To determine the percentage of available chlorine in the given bleaching powder sample.
3. To determine Alkalinity in the given water sample.
4. Qualitative analysis of inorganic mixture.

Cations : NH^{4+} , $Pb^{2+,}$ $Ag^{+,}$ $Bi^{3+,}$ Cu^{2+} , $Cd^{2+,}$ Sn^{2+}, $Fe^{3+,}$ Al^{3+}, $Co^{2+,}$ $Cr^{3+,}$$Ni^{2+,}$$Mn^{2+,}$ $Zn^{2+,}$ $Ba^{2+,}$ $Sr^{2+,}$ $Ca^{2+,}$ K^{+}

Anions : CO_3^{2-} **,** S^{2-}, $SO_3^{2-,}$ $S_2O_3^{2-}$, NO^{3-} , CH_3COO^{-} , Cl^{-} , Br^{-} , I^{-} , NO^{3-} , SO_4^{2-} , $PO_4^{3-,}$ BO_3^{3-}, $C_2O_4^{2-,}$ F^{-}

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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:**

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1.	To determine the chloride content in the given water sample by Mohr‟s method (Argentometric method)			
2.	To determine the percentage of Available chlorine in the given sample of Bleaching powder. Iodometrically			
3.	To determine Alkanlinity in the given water sample.			
4.	To identify the given inorganic compound for its acidic radicals			
5.	To identify the given inorganic compound for its basic radicals			
	Test for zero group			
	Test for zero First group			
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	Test for zero Third group			
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	Test for zero Fifth group			
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Grade Average:

Signature of Teacher:

Name of Teacher:

EXPERIMENT No. 1

Object: To determine the chloride content in the given water sample by Mohr"s method (Argentometric method)

Principle: When silver nitrate solution is added to given sample solution containing few drops of K_2CrO_4 indicator, white silver chloride is precipitated initially. The moment, all the chloride ions have been precipitated out, even a drop of silver nitrate added in excess gives a brick red ppt. of silver chromate. This indicates the end point.

As Ksp (AgCl) < Ksp (Ag_2CrO_4), hence as long as the chloride ions are available, the soluble silver chloride is precipitated. As soon as all the chloride ions have been precipitated out, even a slight excess silver ion produces insoluble silver chromate which is red in colour.

$$AgNO_3 + NaCl \longrightarrow AgCl + NaNO_3$$

$$2AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 + 2KNO_3$$

$$Ag_2CrO_4 + 2Cl^- \longrightarrow 2\,AgCl + CrO_4^{2-}$$

When all the chloride ions are removed as AgCl, than

$$2\,AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 + 2KNO_3$$

Chemicals: N/50 $AgNO_3$, Water sample
Glassware: Burette, Pipette, beaker, conical flask, funnel, glass rod, measuring cylinder etc
Indicator: Potassium chromate (K_2CrO_4)
End point: Yellow color changes to reddish brown color.
Procedure:

1. Take 100 ml of water sample in a conical flask and add 1 to 2 ml of K_2CrO_4 indicator
2. Fill the burette with N/50 AgNO3 solution upto zero mark.
3. Titrate drop by drop along with shaking until the yellow color of solution changes to reddish brown color. This is the end point.
4. Repeat the experiment until the concordant readings are obtained.

Observation Table:

S.No.	Volume of water sample taken (ml)	Burette reading		Concordant Volume of N/50 $AgNO_3$ solution used(ml)
		Initial	Final	

Calculations:

Volume of water sample taken = ___
Concordant Volume of $AgNO_3$ solution used =_____

(i) $N_1 V_1$ = $N_2 V_2$

water sample $AgNO_3$

$N_1 \times 100 = \frac{1}{50} \times X$

$N_1 = \frac{1}{50} \times \frac{X}{100}$

(ii) Strength of Chloride ions = $\frac{1}{50} \times \frac{X}{100} \times \frac{35.5}{1}$ gms/lt

$= \frac{1}{50} \times \frac{X}{100} \times \frac{35.5}{1} \times 1000$ mg/lit.

= ------ ppm

Result: The chloride content in the given water sample is ____ppm

Viva-Voce Questions:

1. What are the sources of chloride ions in natural water?
2. Name the indicator used in this titration?
3. How can you determine the chloride content in the water sample?
4. What is the name of reddish brown ppt. complex formed in this experiment?
5. What do you mean by Argentometric titration?

EXPERIMENT No. 2

To determine the percentage of Available chlorine in the given sample of Bleaching powder. Iodometrically.

Principle: The amount of chlorine liberated by the action of dilute acids on bleaching powder is termed as available chlorine and expressed as percentage weight of bleaching powder. The bleaching powder is a mixture of ($CaOCl_2.4H_2O$)($CaCl_2$ $Ca(OH).H_2O$) and some free $Ca(OH)_2$. When dilute H_2SO_4 reacts with bleaching powder then free chlorine is liberated. The liberated chlorine reacts with potassium iodide to give free iodine. This liberated free iodine is then titrated against N/10 Hypo solution, using freshly prepared starch solution.

$$CaOCl_2 + H_2SO_4 \longrightarrow CaSO_4 + H_2O + Cl_2 \uparrow$$

$$2KI + Cl_2 \longrightarrow 2KCl + I_2$$

$$2Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2NaI$$

Sodium Tetra Thionate

Starch + I_2 ⟶ Starch.Iodide adsorption complex

Deep blue color

Chemicals: N/10 Hypo ($Na_2S_2O_3.6H_2O$),bleaching powder sample solution, solid KI, dilute H_2SO_4.

Glassware: Burette, Pipette, beaker, conical flask, funnel, glass rod, measuring cylinder etc

Indicator: Freshly prepared starch solution.

End point: Disappearance of blue colour.

Procedure:

1. Weigh 5 gm of bleaching powder in clean and pre-weighed weighing tube. Transfer it into a mortar, crush with pestle and add little water.
2. Transfer the paste into 500 ml volumetric flask.
3. Add more water to make solution upto the mark. Shake until a homogenous suspension is obtained.
4. Fill the burette with Hypo solution. Pipette out 25 ml of bleaching powder solution into 250 ml conical flask. Add 2 gm KI and 10 ml dilute H_2SO_4 .Cover the mouth of the conical flask with watch glass and keep in dark for 2 minutes. Solution becomes brown.
5. Titrate liberated Iodine against Hypo solution till a light yellow colour persists.
6. Add 1 to 2 ml of starch solution. Solution turns to blue colour.
7. Continue adding Hypo solution till blue colour disappears. This the end point.
8. Repeat the experiment until the concordant readings are obtained.

Observation Table:

S.No.	Volume of Bleaching powder sample taken (ml)	Burette reading (ml)		Concordant Volume of N/10 Hypo solution used(ml)
		Initial	Final	

Calculations:

Weight of sample taken (W) = 5 gm

Volume of solution prepared = 500 ml

Concordant Volume of N/10 Hypo solution used = ___ml

(i) $N_1V_1 = N_2V_2$

Bleaching Powder — Hypo Solution

$$N_1 \times 25 = \frac{1}{10} \times X$$

$$N_1 = \frac{1}{10} \times \frac{X}{25}$$

(ii) Amount of Chlorine per Litre of solution = y x Equivalent weight

$$= \frac{1}{10} \times \frac{X}{25} \times 35.5 \text{ gms/lt}$$

(iii) Percentage of Available Chlorine =

$$= \frac{1}{10} \times \frac{X}{5} \times \frac{35.5}{1} \times \frac{50}{2} \times \frac{100}{W}$$

Result: The percentage of available chlorine in the given sample of bleaching powder is__

Viva-Voce Questions:

1. What do you mean by available chlorine?
2. What do you mean by iodometric titration?
3. Name the indicator used in iodometric titration.
4. What is hypo? Write down name and formula?

5. What is the chemical name of bleaching powder?
6. Gives the two commercial uses of bleaching powder.

EXPERIMENT No. 3

Object: To determine the alkalinity of given water sample.

Principle: Alkalinity of water may be attributed to the presence of (i) Hydroxide only (ii) Carbonates only (iii) Bicarbonates only (iv) Hydroxide and Carbonates (v) Carbonates and bicarbonates.

The type and extent of alkalinity present in a water sample may be determined by titrating the water sample against a standard acid ,first to phenolphthalein end point (P) and then continuing the titration to methyl orange end point (M).

$$OH^- + H^+ \text{ ------- } H_2O \quad (1)$$
$$CO_3^{2-} + H^+ \text{ ------- } HCO_3^- \quad (2)$$
$$HCO_3^- + H^+ \text{ ------- } H_2O + CO_2 \quad (3)$$

The volume of acid used upto phenolphthalein end point (P) corresponds to the completion of equation (1)and(2) i.e. neutralization of hydroxide (OH^-) and conversion of carbonate (CO_3^{2-}) to bicarbonates (HCO_3^-) takes place at this stage, while the volume of acid used after (P) corresponds to the completion of equation (3) i.e. neutralization of bicarbonates, HCO_3^- takes place.

The total amount of acid used from the beginning of the experiment i.e (M) corresponds to the total alkalinity and represents the completion of reaction shown by equation (1) to (3). **Chemicals required:** Water sample, N/50 H_2SO_4

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

Indicators:

Phenolphthalein (pH- 8.3 to 10).

Methyl orange (pH- 3.1 to 4.4).

End point: For Phenolphthalein- Pink color changes to colorless.

For Methyl orange - Light yellow color changes to light red.

Procedure:

1. Take 25 ml of water sample in a conical flask and add 2 or 3 drops of phenolphthalein indicator.
2. Titrate this sample against N/50 H_2SO_4 solution with continuous shaking until the pink color just disappears.
3. Note down the reading from the burette which corresponds to phenolphthalein end point (P)
4. Now add 2 drops of methyl orange indicator to the same solution, continue the titration with N/50 H_2SO_4 solution till yellow color changes to light red.
5. Note the volume of H_2SO_4 used from burette which corresponds to methyl orange end point (M).
6. Repeat the experiment to get two more readings.

Observation table:

S.No.	Volume of water sample taken (ml)	Burette reading Volume of N/50 H_2SO_4 solution used		
		Phenolphthalein end point (ml)		Methyl orange endpoint (ml)
		Initial	Final	
1.				
2.				
3.				

Calculations:

Volume of water sample taken =……ml.

Volume of H_2SO_4 used to phenolphthalein endpoint (P) = ----- ml.

Volume of H_2SO_4 used to methyl orange endpoint (M) = ----- ml.

$$N_1V_1 = N_2V_2$$

(water sample) (H_2SO_4)

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

Strength in terms of $CaCO_3$ = $\frac{1 \times a \times 50}{50 \times V_1}$ g/L

Carbonate alkalinity = $\frac{1 \times 2a \times 50 \times 1000}{50 \times V_1}$ mg/L

1 mg/L = 1ppm = ____ppm (parts per million)

Bicarbonate alkalinity = $\frac{1 \times (b-2a) \times 50 \times 1000}{50 \times V_1}$ mg/L

=____ppm

Total alkalinity = $\frac{1 \times b \times 50 \times 1000}{50 \times V_1}$ mg/L

= ……ppm

(i) Carbonate alkalinity = ……ppm
(ii) Bicarbonate alkalinity = ……ppm
(iii)Total alkalinity = ……ppm

Viva-Voce Questions:

1. What do you mean by Alkalinity?
2. Which ions are responsible for the alkalinity of water?
3. Name of the indicator used in this experiment?
4. What will be the colour of phenolphthalein and methyl orange indicator in acidic and basic medium?
5. Why hydroxide ions and bicarbonate ions cannot be mix together?
6. Name of the type of titration used in the estimation of alkalinity in water?

Scheme for Systematic Analysis of a Mixture containing two Salts

Qualitative Analysis

EXPERIMENT No. 4(a)
Tests For Anions

Object:- To identify the given inorganic compound for its acidic and basic radical.

Chemicals : Dilute H_2SO_4, Lime water ,$K_2Cr_2O_7$, lead acetate solution, neutral $FeCl_3$,NH_4OH, dilute CH_3COOH ,dilute nitric acid ,silver nitrate solution, starch solution, $FeSO_4$ solution

Apparatus: Test Tube, Test Tube holder, Spatula, Burner , filter paper, starch iodide paper

Acidic Radical

Group A: CO_3^{2-}, , **SO_3^{2-} , S^{2-}, NO_2^-,CrO_4^{2-}**

Group B: Cl^-, Br^-, I^-, NO_3^-, CH_3COO^-, C_2O4^{2-}

Group C: SO_4^{2-}, **PO_4^{3-}**

Preparation of Sodium Carbonate Extract:
Mix 100 mg of the given salt with 250 mg of anhydrous Na_2CO_3 and add about 10 to 20 ml of distilled water and boil it for about 15 minutes. The mixture is then cooled and filtered . The filterate is known as Sodium Carbonate Extract. It is used to perform wet tests.
Use it for conforming sulphide, sulphite, sulphate, chloride, bromide, iodide, oxalate and phosphate.

(a) **Dilute Sulphuric acid test**
To a little of the mixture in a test tube add few drops of dilute H_2SO_4 .Observe the colour and smell of evolved gas in cold and then after warming the contents in a water bath.

S.No.	Observation	Inference	Confirmatory Test
1.	Colourless gas with brisk effervescence evolved.	Carbonate (CO_3^{2-}) may be present.	Pass the above evolved gas through lime water. Lime water turns milky. **Carbonate (CO_3^{2-})confirmed**
2.	Colourless gas with smell of burning sulphur evolved.	Sulphite (SO_3^{2-})may be present.	Place a filter paper moistened with acidified $K_2Cr_2O_7$ over the mouth of the test tube. It is turned green. **Sulphite (SO_3^{2-})confirmed**
3.	Colourless gas with smell of rotten eggs evolved.	Sulphide may be present.	Place a filter paper moistened with lead acetate solution over the mouth of the test tube. It is turned black. **Sulphide confirmed**.
4.	Reddish brown gas with pungent smell evolved.	Nitrite (NO_2^-) may be present.	Expose an acidified starch iodide paper over the mouth of the test tube. It is turned black blue.

			Nitrite (NO_2^-) confirmed.
5.	Colourless gas with a characteristic odour	Acetate (CH_3COO^-) may be present	To 5-6 drops of the solution of the substance add 1- 2 drops of neutral $FeCl_3$ solution. A blood red colour. **Acetate (CH_3COO^-) may be present**

Concentrated Sulphuric acid test :

To a little of the mixture in a test tube add few drops of conc. H_2SO_4 and warm the test tube in a water bath for a minute. Observe the colour and smell of evolved gas and make inferences.

S.No.	Observation	Inference	Confirmatory Test
1.	Colourless gas with pungent smell evolved.	Chloride **(Cl^-)** may be present.	(i) Bring a glass rod moistened with NH_4OH at the mouth of the test tube. White dense fumes of NH_4 Cl are formed. (ii) Chromyl chloride test .Add few mg of potassium dichromate crystals and 5 drops of conc. sulphuric acid and heat. Passed the vapours through the test tube which contains sodium hydroxide solution. To this yellow solution, add dilute CH_3COOH and lead acetate solution. Yellow coloured precipitate is formed. **Chloride confirmed**.
2.	Brown colour gas evolved.	Bromide (Br^-) may be present.	Silver nitrate test . Acidify 2 or 3 drops of soda extract with dilute nitric acid and1-2 drops of silver nitrate solution . A pale yellow precipitate formed. **Bromide confirmed**
3.	Violet colour gas evolved.	Iodide (I^-) may be present.	Hold a filter paper strip moistened with starch solution over the mouth of the test tube. It is turned blue. **Iodide confirmed.**
4.	Light brown colour gas evolved.	Nitrate (NO_3^-) may be present.	(i)Add copper turnings. Brown colour intensifies. (ii) Brown Ring Test. The soda extract is mixed with 2 or 3 drops of dilute H_2SO_4 and 1or 2 drops freshly prepared $FeSO_4$ solution along the sides of the test tube without shaking. Brown ring is formed at the junction of the two liquids.

			Nitrate (NO_3^-) confirmed.
5.	Colourless gas turns lime water milky	Oxalate ($C_2O_4^{2-}$) may be present.	$CaCl_2$ test . Acidify 5 drops of soda extract with acetic acid and add1 ml of $CaCl_2$ solution. A white ppt appears within a minute.Centrifuge and separate the ppt. Dissolve the ppt in 10 drops of dilute H_2SO_4 .Heat in a water bath. Add 1-2 drops of $KMnO_4$ solution. Pink colour disappears. **Oxalate ($C_2O_4^{2-}$) confirmed**

Viva-Voce Questions:

1. What are Radicals?
2. What are a cations and anions?
3. What do you mean by acid and basic radicals?
4. Name the radicals which are detected by dilute acid.
5. Name the radicals which are detected by conc.H_2SO_4 test.
6. Which gas is evolving by the action of dilute acid on carbonate?
7. What is lime water?
8. What happen when CO_2 is passed through lime water?
9. How will you prepare soda extract?
10. Distinguished between soda extract and sodium extract.

Scheme for Systematic Analysis of a Mixture containing two Salts

Qualitative Analysis

EXPERIMENT No. 4(b)
Tests For Cations

Object: To identify the given inorganic compound for its acidic and basic radical.

Chemicals :

Apparatus:, Test Tube, Test Tube holder, Spatula, Burner

Basic Radical:

Group 0 NH_4^+

Group 1 Pb^{2+} , Ag^+

Group 2 Pb^{2+}, Cu^{2+}, As^+

Group 3 Fe^{3+}, Al^{3+}, Cr^{3+}

Group 4 Mn^{2+}, Zn^{2+}, Ni^{2+}, Co^{2+}

Group 5 Ca^{2+}, Ba^{2+}, Sr^{2+}

Group 6 Mg^2

Test for Basic Radicals

Preparation of Original Solution .

Take about 10 mg of the given substance in a boiling tube The mixture should be dissolved in a suitable solvent first in cold and then by heating for few minutes. The choice of solvents can have the following sequence.

1. Water 2. Dilute HCl 3. Conc. HCl, 4. Dilute HNO_3, 5 Conc. HNO_3, and 6. Aquaregia.

The solution prepared by dissolving the mixture in the solvent is referred to as the original solution or O.S.

Generally conc. HCl is used for preparing solutions.

Test for Zero group

S.No.	Experiment	Observation	Inference
1	Heat a little of the OS with sodium hydroxide.solution .	Ammonical smelling gas evolved.	Ammonium NH_4^+ may be present
2	To one ml of aqueous mixture of solution add few drops of Nessler's ($K_2[HgI_4]$) reagent and warm	A brown colour or precipitate is obtained.	Ammonium NH_4^+ confirmed.

Test for First group

Members: Ag^{+} Pb^{2+} , Hg_2^{2+}

Procedure: To the original solution (OS) in any solvent other than HCl add dil. HCl drop by drop. If a ppt is formed, continue adding HCl until no further precipitation takes place. Filter the ppt. Keep the filtrate for the analysis of subsequent groups and examine the ppt. for the members of first group.

Examine the precipitate of Group 1

The white ppt obtained on adding dil. HCl to the original solution may consist of $PbCl_2$,AgCl, and Hg_2Cl_2. Wash the precipitate on the filter paper with cold water and reject washings. Transfer the ppt. to a boiling tube and add 10 – 15 ml of distilled water. Boil for few minutes and filter while hot.

Residue. May contain Ag^{+} &. Hg_2^{2+}
Wash the ppt on the filter paper with hot water until the washings give no ppt. with K_2CrO_4 solution. Reject washings. Pour 10 -15 ml of warm dilute NH_4OH over the ppt and collect the filtrate.

Filtrate. May contain Ag^{+}
Divide the filtrate into two parts.

- Acidify one part with excess of dil.HCl. or dil. HNO_3.
  A white curdy ppt is formed.
- To other part add KI solution .
  A light yellow ppt. is formed

Silver(Ag^{+}) present

Filtrate. May contain Pb^{2+}.
Divide the filtrate into three parts.

- Cool one part under tap. Needle shaped white crystal of $PbCl_2$ separate out.
- Add few drops of K_2CrO_4 solution.
  Yellow precipitate due to the formation of $PbCrO_4$ soluble in NaOH but insoluble in acetic acid
- To another part add KI solution.
  Yellow precipitate due to the formation of PbI_2 is formed which is soluble in hot water and reappears on cooling.

Lead(Pb^{2+}) present

Test for Second group

Members: (Hg, Pb, Cu, As, Sb, Sn)

Procedure: To the original solution (OS) add dil. HCl drop by drop. If no ppt is formed, first group is absent. Proceed directly to second group.

Pass H_2S gas to the OS prepared in dil. HCl 0r to the filtrate of first group till complete precipitation. Wash the ppt. 2 or 3 times with hot water .Punch a hole through the filter paper and wash down the ppt with 5 ml of yellow ammonium sulphide and some NaOH solution in a beaker. Heat in boiling water bath for five minutes and filter.

<table>
<tr>
<td>Residue May contain II A sub group (HgS , PbS, CuS etc)
Wash the residue with 2 or 3 times with hot water until free from yellow colour. Transfer the ppt. to a test tube and boil with about 5 ml of dil. HNO_3 for few minutes. Filter and collect it the filtrate.
Filtrate (A) . May contain Cd & Cu.
Add excess of NH_4OH to the filtrate. Warm and filter.
Filtrate (B). Blue . Acidify with 4 0r 5 drops of acetic acid and add $K_4[Fe(CN)_6]$.
A chocolate brown ppt is obtained.

Copper (Cu^{2+}) present</td>
<td colspan="3">Filtrate May contain II B sub group(As, Sb, Sn)
Add dil. HCl until acidic to litmus. Warm gently.
A yellow or orange ppt indicates II B group. Filter the ppt. and reject the filtrate. Boil this ppt. with 10 ml of conc. HCl for five minutes and filter it.</td>
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<tr>
<td></td>
<td>Residue. May contain Arsenic (As^{3+})</td>
<td colspan="2">Filtrate. May contain $SbCl_3$ and $SnCl_4$. Add NH_4OH to make the filtrate just alkaline to litmus paper. Add4-5 gm of oxalic acid and boil.Pass H_2S and filter.</td>
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<tr>
<td></td>
<td></td>
<td>Residue. May contain Antimony (Sb^{3+})</td>
<td>Filtrate. Add ammonium molybdate solution .
A deep blue colour or ppt. is formed.

Tin (Sn^{2+} or Sn^{4+}) present</td>
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</table>

Test for Third group

Members: (Fe^{3+}, Al^{3+}, Cr^{3+})

Procedure: Take the filtrate of IInd group and boil to remove H_2S completely. Add few drops of conc. HNO_3 and evaporate nearly to dryness. Test for interfering radicals like phosphates, oxalate, borate and fluoride. If present, remove them. Proceed with the further analysis as below.

Take the above solution (already boiled with conc. HNO_3), add 1 gm of solid NH_4Cl and heat and then add NH_4OH until in excess. If a ppt. is obtained III **rd** group is present. Heat the solution and filter immediately. Preserve the filtrate for the analysis of subsequent groups and examine the ppt. for the members of III **rd** group.
The ppt. may contain $Fe(OH)_3$, $Cr(OH)_3$, $Al(OH)_3$ and a little $MnO_2.xH_2O$. Wash the ppt. with hot water, transfer it to a boiling tube and dissolve in minimum quantity of dilute HCl. And add excess of NaOH, boil and filter.

Filtrate. May contain Al^{3+} . Add 1 gm solid NH_4Cl and boil. A white gelatinous ppt . of $Al(OH)_3$ **Aluminium (Al^{3+}) present**	**Residue .** May contain $Fe(OH)_3$, $Cr(OH)_3$, $Al(OH)_3$ and a little $MnO_2.xH_2O$. Perform test for Fe, Cr, and Mn in this residue.		
	Iron. Dissolve a small portion of residue in dil. HCl and divide into two parts. (i) To one part add $K_4[Fe(CN)_6]$ solution. A Prussian blue colour or ppt. is formed. (ii)To second part add KCNS solution . A blood red is formed. **Iron (Fe^{3+}) present**	**Chromium.** Boil a little of the residue with NaOH and excess of Br_2 water and filter . If the filtrate is yellow.Cr^{3+} may be present. To the filtrate add excess of acetic acid and lead acetate solution. A yellow ppt. of $PbCrO_4$ indicates Cr^{3+} **Chromium (Cr^{3+}) present**	**Manganese.** Dissolve a small portion of residue in dil. HNO_3.Add 1 gm of lead di-oxide and boil. Allow to stand for a few minutes. A purple is obtained. **Mn^{2+} of IV th group present**

Test for Fourth group

Members: Zn^{2+}, Mn^{2+}, Ni^{2+}, and Co^{2+}

Procedure: To the filtrate of **IIIrd** group (already contain NH_4Cl) add NH_4OH until it gives smell of ammonia. Warm and pass H_2S .If a ppt. is obtained , IVth group is indicated. Continue passing H_2S until the precipitation of IVth group is complete. Heat and filter. Preserve the filtrate for the analysis of subsequent groups and examine the ppt. for the members of IVth group.

The ppt. may contain ZnS (white), MnS (white), NiS (black).Wash the ppt. with hot water and transfer it to a small beaker. Add about 10 ml of *very dil. HCl* (1:10).Shake well, warm a little and filter.

<table>
<tr>
<td>Residue. Black may contain NiS and CoS.
Dissolve the residue in 5 or 6 drops of aqua regia in a boiling tube and boil gently to expel chlorine. Add 0.5 ml of distilled water and divide into two parts.
<u>Test for Nickel:</u> To one part add conc. NH_4OH to alkaline it and then add 4 0r 5 drops of dimethyl glyoxime.
A red ppt. confirms the nickel.

<u>Test for Cobalt:</u> To second part add 0.5 ml of acetone and 50 mg ofsolid NH_4CNS and shake. Blue colour in acetone layer confirms cobalt.</td>
<td colspan="2">Filtrate: May contain $ZnCl_2$ and $MnCl_2$ .Boil the solution to remove H_2S (test the vapours with lead acetate paper).Cool and add excess of NaOH solution. Boil and filter.

Residue. Brown May contain Mn^{2+}.
Dissolve the residue in conc. HNO_3. Add 1 gm lead peroxide and boil and allow the solution to settle down.
A violet pink colour

Manganese (Mn^{2+}) present</td>
<td>Filtrate. May contain Zn^{2+} .
Add acetic acid and $K_4[Fe(CN)_6]$ solution.
A bluish-white ppt of $Zn_2[Fe(CN)_6]$ is obtained

Zinc(Zn^{2+}) present</td>
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</table>

Test for Fifth group

Members: (Ba^{2+}, Sr^{2+}, Ca^{2+})

Procedure: Take the filtrate of IVth group in a beaker and acidify with acetic acid and boil off H_2S completely. Concentrate the solution by evaporation to a small bulk (10 ml). Add NH_4OH to make it alkaline and then add freshly $(NH_4)_2CO_3$ in slight excess. Warm upto 60° C and allow to stand for 2-3 minutes and filter. Preserve the filtrate for VI groupth and examine the ppt. for Vth group.

Wash the ppt. with hot water and dissolved in 1 ml of dil. acetic acid and warm. Take 2-3 drops of this solution and test this solution for barium by adding 1-2 drops of K_2CrO_4 solution.

A yellow ppt. indicates Ba^{2+}

If barium is absent, throw the test solution and test for Sr and Ca in the remaining solution and treat it as filtrate (a)

<table>
<tr><td colspan="2">Filtrate (a) May contain $Sr^{2+}$and Ca^{2+} . Add $(NH_4)_2SO_4$ solution in excess and boil.Scratch the sides of the est tube and wait for 5 minutes. A white ppt. is formed. Filter.</td></tr>
<tr><td>Residue. White ppt. confirms Strontium
Apply flame test as in Ba. Crimson red flame

Strontium (Sr^{2+}) present</td><td>Filtrate. May contain Calcium (Ca^{2+}).
Add a little ammonium oxalate solution and warm.
A white ppt. of calcium oxalate
(i) Dissolve the ppt. in dilute H_2SO_4 and add few drops of $KMnO_4$ solution.
Pink colour of $KMnO_4$ solution is decolorized.

Calcium (Ca^{2+}) present</td></tr>
</table>

Test for sixth group

Add 0.5 ml of conc.HNO_3 to the centrifugate of V group and evaporate to dryness. Dissolve the residue in 1 ml of distilled water.

Magnesium	**Sodium**	**Potassium**
1) To 2 drops of above solution or the filtrate of V group,add 2 drops of titan yellow solution and one drop of NaOH solution.A red coloure or ppt is obtained.	1) To 3-4 drops of above solution ,add 3 drops potassium pyroantmionate and 2 drops of dil.acetic acid.Warm gently.A white ppt.obtained on cooling.	1)To 3-4 drops of above solution,add 3-4 drops of sodium cobalt nitrite solution.A yellow ppt is obtained.
2) T o drops of the above solution or filtrate of V group, add 2 drops of magneson reagent and 2 drops of NaOH.A blue ppt is obtained. Magnesium (Mg) is present.	2) Apply flame test. Sodium (Na) is present.	2) Apply flame test. Potassium (K) is present.

Viva-Voce Questions:

1. What is a group reagent?
2. What is Nessler's reagent?
3. Can we use sulphuric acid to prepare original solution for cation analysis?
4. Why H_2S turn lead acetate paper black.
5. If the mixture dissolves in hot HCl and on cooling, a white ppt is formed, what is the reason for it?
6. What is the function of HCl in second group?
7. What is the function of conc. HNO_3 in third group analysis?
8. What is the function of NH_4Cl in fourth group analysis?
9. What is the function of NH_4Cl & NH_4OH in fifth group analysis?
10. How will you prepare H_2S gas?

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