SVR ENGINEERING COLLEGE

(Ayyaluru Metta(V), Nandyal)
(Affiliated to JNTUA, Ananthapuramu & Approved by AICTE, New Delhi)

ANDHRA PRADESH-518502

2021-22

CHEMISTRY LABORATARY MANUAL

(20A51101P) - CSE, AI, ECE & EEE

Prepared by

Dr. S SREENIVASULU Associate Professor
Mr M SHANOOR BASHA Assistant Professor
Mr. M SREENIVASULU Assistant Professor
Mr P PARABRAHAMAM Assistant Professor
Ms P HIMA BINDU Assistant Professor

for

I B.TECH

DEPARTMENT OF HUMANITIES & SCIENCE

CERTIFICATE

This is to certify that Mr./Miss…….……...

Registered No……..……..…… has successfully completed the Experiments provided in the Applied Physics Laboratory developed by the Department of Physics, SVR Engineering College, Nandyal prescribed for the First Year B.Tech Courses, and as Approved by the Jawaharlal Nehru Technological University, Anantapur for the year …….

(Signature)
Head of the Department

(Signature)
Faculty In-charge

STUDENT PROFILE

Name of the Student : ----------

Regd. Number : ----------

Branch / Section : ----------

Contact Phone No : ----------

E-mail ID : ----------

Father's Name : ----------

Father's Contact No : ----------

Residential Address : ----------

VISION OF THE COLLEGE

To produce competent engineering graduates & Managers with a strong base of Technical & Managerial knowledge and the complementary skills needed to be successful professional engineers & Managers.

MISSION OF THE COLLEGE

To Fulfill The Vision By Imparting Quality Technical & Management Education To The Aspiring Students, By Creating Effective Teaching / Learning Environment And Providing State-Of The –Art Infrastructure And Resources.

DEPARTMENT OF HUMANITIES AND SCIENCES

VISION

To build foundation for excellence and spur development of the Institution as a premier Institution by igniting and nurturing enthusiasm, interests and passion in the study of Mathematics, Physics, Chemistry & English in professional courses, as a part of curriculum.

MISSION

- To awaken the young minds and discover their talents both in theory and in practical Physics through dedicated teaching, commitment to students and innovative instructional methods.
- To support the developmental activities of the College and make the department vibrant.
- To make vital contributions in areas of emphasis such as faculty, modern labs, Department library and demonstrate a high level of competence in the study of Mathematics, Physics, Chemistry & English
- To introduce pioneering programs in the Department that will embrace heritage and values of the Institution.
- To organize and sustain efficient operating systems in the Department for realization of our objectives as Institution of eminence and International standards.

To evolve strategies in the Department for continuous Improvement. **PROGRAMME OUTCOMES**

By the end of completing 4 years full time B.Tech in CSE, Graduates of B.Tech will be able to:

PO 1.Engineering Knowledge: Apply the knowledge of mathematics, science, engineering fundamentals, and an engineering specialization to the solution of complex engineering problems.

PO 2. Problem Analysis: Identify, formulate, review research literature, and analyze complex engineering problems reaching substantiated conclusions using first principles of mathematics, natural sciences, and engineering sciences.

PO 3.Design/Development of Solutions: Design solutions for complex engineering problems and design system components or processes that meet the specified needs with appropriate consideration for the public health and safety, and the cultural, societal, and environmental considerations.

PO 4. Conduct Investigations of Complex Problems: Use research-based knowledge and research methods including design of experiments, analysis and interpretation of data, and synthesis of the information to provide valid conclusions.

PO 5. Modern Tool Usage: Create, select, and apply appropriate techniques, resources, and modern engineering and IT tools including prediction and modeling to complex engineering activities with an understanding of the limitations.

PO 6. The Engineer and Society: Apply reasoning informed by the contextual knowledge to assess societal, health, safety, legal and cultural issues and the consequent responsibilities relevant to the professional engineering practice.

PO 7. Environment and Sustainability: Understand the impact of the professional engineering solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.

PO 8.Ethics: Apply ethical principles and commit to professional ethics and responsibilities and norms of the engineering practice.

PO 9. Individual and Team Work: Function effectively as an individual, and as a member or leader in diverse teams, and in multidisciplinary settings.

PO 10. Communication: Communicate effectively on complex engineering activities with the engineering community and with society at large, such as, being able to comprehend and write effective reports and design documentation, make effective presentations, and give and receive clear instructions.

PO 11. Project Management and Finance: Demonstrate knowledge and understanding of the engineering and management principles and apply these to one's own work, as a member and leader in a team, to manage projects and in multidisciplinary environments.

PO 12. Life-long Learning: Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change.

Engineering Chemistry Lab Manual

1) *Introduction*

The On-Line Lab Manual serves as your text for the lab portion of the courses (B.Tech., B.Sc.& M.Sc.(Industrial Chemistry). 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:*

Personal Protective Equipment (PPE) and Safe Attire

a. Wear chemical safety goggles and a knee length (41-42 inch) laboratory white coat at alltimes 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 contactwith chemicals.

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 drinkssealed 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

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. 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.

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 wastechemicals. 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.

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

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.

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 appropriatechemical hygiene policies and practices;
2. Monitor procurement and use of chemicals in the lab, determining that laboratory facilitiesand 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, 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

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.

A. Chemical Handling

Each laboratory employee (with training, education, and resources provided by supervision) shalldevelop 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 allchemicals 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 shallbe implemented as deemed necessary by the CHP.

B. 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 flushingshall 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.

C. 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.

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.

Laboratory Coats:

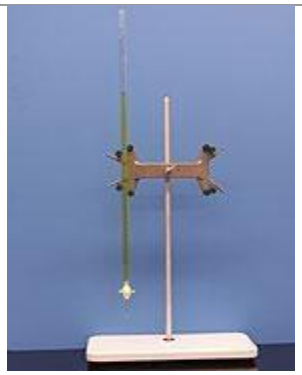
1. Purchase a lab coat that fits you well. Lab coats that are too tight or too loose are not safe.Sleeves that are too long should be rolled up.
2. If your lab coat has not been contaminated with a hazardous substance, you may wash it as you do your other clothing.
3. If your lab coat becomes contaminated 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.

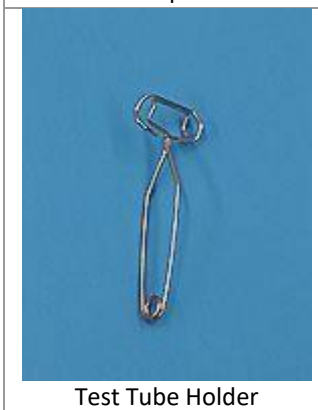
Nitrile Gloves:

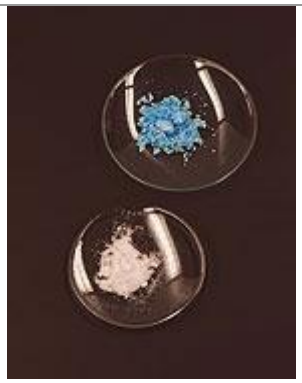
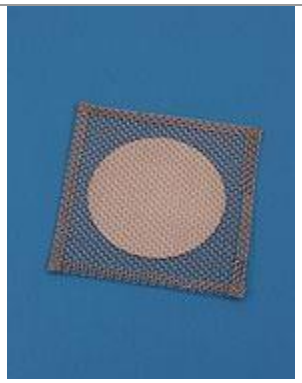
a) Nitrile gloves are to be worn only during portions of experiments where specified by the experimental procedure, when instructed by the instructor or supervisor, or when workingwith substances for which the protocol requires the use of gloves.
b) Note that nitrile gloves are flammable and will stick 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 inthe 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.

Chemistry Laboratory Common Instruments:

Below are photos and names of common lab equipment you will encounter in Chemistry lab listed inalphabetical 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

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/orby 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.

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.

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 thequantity 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 solutionof 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.

Thestrength 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)

INDEX

JNTU SYLLABUS

S.No.	Name of the Experiment
1	Measurement of 10Dq by spectrophotometric method.
2	Models of potential energy surfaces.
3	Conductometry titration of (i) strong acid vs. strong base, (ii) weak acid vs. strong base.
4	Determination of cell constant and conductance of solutions.
5	Potentiometry - determination of redox potentials and emfs.
6	Determination of Strength of an acid in Pb-Acid battery.
7	Preparation of a Bakelite and measurement of its mechanical properties (strength.).
8	Verify Lambert-Beer's law.
9	Thin layer chromatography.
10	Identification of simple organic compounds by IR.
11	Preparation of nonmaterial's by precipitation.
12	Estimation of Ferrous Iron by Dichrometry.

Chemistry laboratory outcomes (20A51101P)

S. No	LAB outcomes		Cognitive Process Dimension
1	**CO1**	Determine the cell constant and conductance of solutions	**Apply**
2	**CO2**	Prepare advanced polymer materials	**Understanding**
3	**CO3**	Determine the physical properties like surface tension, adsorption and viscosity	**Apply**
4	**CO4**	Estimate the Iron and Calcium in cement	**Apply**
5	**CO5**	Calculate the hardness of water	**Analyzing**

LIST OF EXPERIMENTS TO BE CONDUCTED

S.No.	Name of the Experiment
1	Measurement of 10Dq by spectrophotometric method.
2	Models of potential energy surfaces.
3	Conductometrictitration of (i) strong acid vs. strong base, (ii) weak acid vs. strong base.
4	Determination of cell constant and conductance of solutions.
5	Potentiometry - determination of redox potentials and Emfs.
6	Determination of Strength of an acid in Pb-Acid battery.
7	Preparation of a Bakelite and measurement of its mechanical properties (strength.).
8	Verify Lambert-Beer's law.
9	Thin layer chromatography.
10	Identification of simple organic compounds by IR.
11	Preparation of nonmaterial's by precipitation.
12	Estimation of Ferrous Iron by Dichrometry.

SCHEME OF VALUATION

Internal Lab Exam

1.Day today Observation : 10 M

2. Day to day performance : 10M

3.Day today Record : 10 M

Final Internal Lab Exam : 30 M

Total Internal Marks : 30 M

External Lab Exam

1. Record : 10 M

2. Write up : 25 M

3. Experiment : 15 M

4. Result : 10 M

5. Viva-Voce : 10 M

Total External Marks : 70 M

Total Final Lab Marks : 70 M

WRITE UP PROCEDURE

1. FORMULA WITH TERMS EXPANSION
2. MODEL GRAPH
3. PROCEDURE
4. TABULAR FORMS
5. PRECAUTIONS
6. CALCULATIONS
7. RESULT

Rules to be followed in Chemistry Laboratory

1. Student should wear Apron whenever they enter the laboratory for practice.
2. Student should wear Identification card (ID).
3. Student should come to the lab along with Observation book and Record.
4. While entering and leaving the lab, student should sign in the log book.

Experment-1
Measurement of 10Dq by spectrophotometric method

Aim: To Measure 10 Dq for transition metal complexes by spectrophotometric method.

Chemicals: $CrCl_3.6H_2O$, $Cr(H_2O)_6(NO_3)_2.3H_2O$, Charcoal, Ethylenediamine, Ethanol, Hydrochloric acid, Acetyl acetone, Urea, Benzene, Hexane or Heptane.

Apparatus: Beaker, Steam bath, UV-Visible spectrophotometer

Preparation of Triethylendiaminechrmium(III)Chlmide

The method of preparation of the complex in its hydrated form is essentially and proceeds as follows: To 6.7 g $CrCl_3.6H_2O$, which has been thoroughly ground in a mortar and pestle, add 0.8 g decolorizing charcoal (catalyst) and 9.5 g ethylenediamine. Heat mixture in an open beaker on asteam bath overnight. After cooling, cover with 25 ml ethanol and, after an hour, grind mixture in a mortar, filter off alcohol, and repeat several times with smaller portions of ethanol to remove all excess ethylenedismine (one need not wait an bow in these steps). After drying, stir mixture with 25 ml of 1.7 N HCl at 60° for one minute, filter rapidly with a Biichner filter, and add the filtrate immediately to aniee-cooled mixture of 40 mi ethanol and 25 ml cone. HC1, the ethlmol-HCI mixture being contained in a beaker. The orange $Cr(en)Cl_3$ 3.5 H_2O which separates is filtered out, washed with ethanol,and air dried.

Preparation of Tris(2,4-pentanedionato)ehronmiun1(III)

The methodof preparation, 'To 100 ml H.0 are added 2.66 g of $CrCl_3.6H_2O$ (0.01 mole)and, after complete solution, 20 g of urea and 6 g of acetyl acetone (0.06 mole). The reaction mixture is covered with awatch glass and heated on a steam bath overnight. As theurea hydrolyzes to release ammonia, purple crystals of thecomplex form. Recrystallize the crude product by dissolvingin a minimum quantity of benzene, filtering, and adding hexaneor heptane to precipitate the $Cr(C_5H_7O_2)_3$.

Discussions': the above prepared complexes are soluble in water and benzene, find the energy gap between electronic energy levels using UV-visible spectrophotometer method. The energy gap is identical to the 10Dq, which gives experimentally without any calculation. The above complexes draw the graph between Molar absorbtivity or absorbance Vs wavelength. The graph shown below. And the 10Dq for the above complexes given in Table.

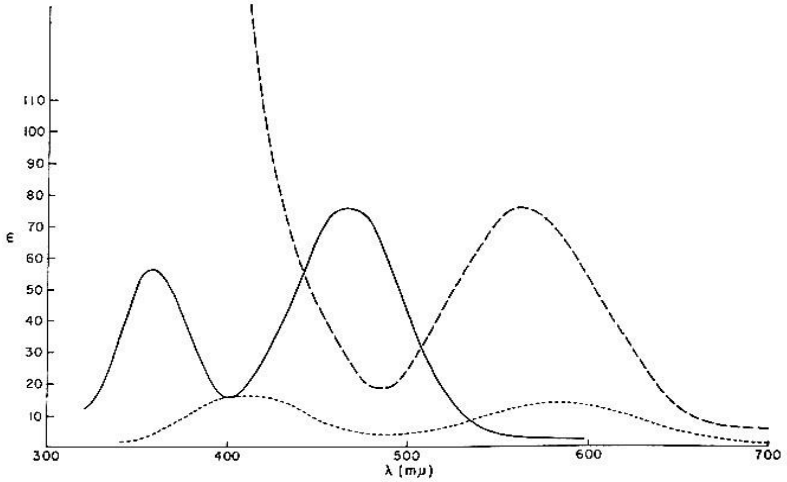


Fig: The absorption spectrum of $Cr(en)Cl_3$ 3.5 H_2O in water [solidlinel, the spectrum of $Cr(H_2O)_6(NO_3)_2.3H_2O$in wotcr (dotted line) and thespecbum of $Cr(C_5H_7O_2)_3$ in benzene (broken line).

Table: Experimental Values of 10 Dq of Cr(lll) Complexes

Chromium (III) complex	**10Dq Cm^{-1}**
$Cr(en)Cl_3$ 3.5 H_2O	21,300
$Cr(C_5H_7O_2)_3$	17,750
$Cr(H_2O)_6(NO_3)_2.3H_2O$	17,100

Result: the above complexes measured 10dq by using spectrophotometric method.

Experiment-2

Hydrogen-Bonded Base Pairs (potential energy surfaces)

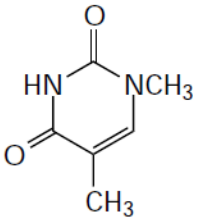
Aim : To model hydrogen-bonding between DNA base pairs in terms of charge-charge interactions. Toidentify nucleotide mimics.

Background:

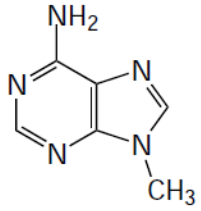
The genetic code is "read" through the selective formation of hydrogen-bonded complexes, or Watson-Crick base pairs, of adenine (A) and thymine (T) (forms A-T base pair), and guanine (G) and cytosine(C) (forms G-C base pair). The importance of this hydrogen bond-based code lies in the fact that virtuallyall aspects of cell function are regulated by proper base pair formation, and many diseases can be tracedto "reading" errors, i.e., the formation of incorrect base pairs.

Procedure:

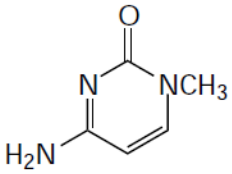
Calculate geometries, energies and electrostatic potential maps for **MeT**, **MeA**, **MeC** and **MeG**.



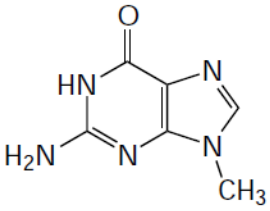
1-methylthymine
(MeT)



9-methyladenine
(MeA)



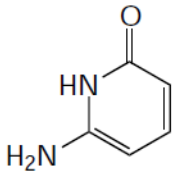
1-methylcytosine
(MeC)



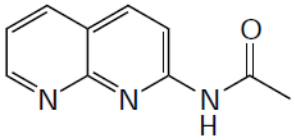
9-methylguanine
(MeG)

Identify electron-rich and electron-poor sites that would be suitable for hydrogen bonding (consider only sites in the plane of the molecules). Draw all possible base pairs that involve two or three hydrogen bonds (do not limit yourself to Watson-Crick pairs). Choose one system (your best guess for the most favorable base pair involving two hydrogen bonds or most favorable base pair involving three hydrogen bonds). Obtain the geometry and energy of this base pair and calculate the total hydrogen bond energy.

Calculate the geometries, energies and electrostatic potential maps for **AP** and **NA**, heterocyclic moleculesthat mimic the hydrogen bonding properties of DNA nucleotides.



6-amino-2-pyridone
(**AP**)



N-naphtharidinyl acetamide
(**NA**)

Identify electron-rich and electron-poor sites for each to see if either would be a suitable partner for one of the bases in your chosen base pair. Obtain the geometry and energy of the resulting hydrogen-bonded adducts and calculates the total hydrogen bond energy.

Result: Physical properties of Hydrogen-bonded bases pairs were calculated using potential energy diagrams.

Experment-3
CONDUCTOMETRIC TITRATION

STRONG ACID VS STRONG BASE

Aim: To measure the equivalent point between strong acid and strong base using conductometer

REQUIREMENTS

Chemicals: Hydrochloricacid, Sodium hydroxide, Distilled water

Apparatus: Conductometer, Conductivity cell, Burette, Pipette, Beaker (100ml)

THEORY:

Neutralization between a strong acid (HCl) and a strong base (NaOH) is represented by

$$H^+ + Cl^- + Na^+ + OH^- \rightarrow Na^+ + Cl^- + (H_2O)$$

It is evident from the above equation that as NaOH solution is gradually added , the H^+ ion having high ionic conductance are replaced by Na^+ having lower ionic conductance and hence the conductivity of the solution gradually decrease. At the equivalent point the conductivity would be minimum due to replacement of all H^+ ion by Na^+ ion. After the equivalence point Na^+ and OH^- would accumulate in the solution and conductance of the solution will again increase. If the conductance corresponds to the volume of NaOH solution added be plotted, two straight lines having opposite slopes would be obtained. The point of intersection of the two straight lines would give the equivalence point.The strength of NaOH solution should be at least 5 times greater than that of the HCl solution, so that the effect of volume change on the conductance be negligible.

CONDUCTOMETRIC TITRATION:

20ml of supplied HCl solution (strength in the order of N\10) is pipette out into a 250ml beaker and about 100ml of distilled water is added. The burette was filled with NaOH solutions and number of drops of NaOH solutions corresponding to a measured volume was calculated .From this calculation 1 drops corresponding to how much volume is estimated.

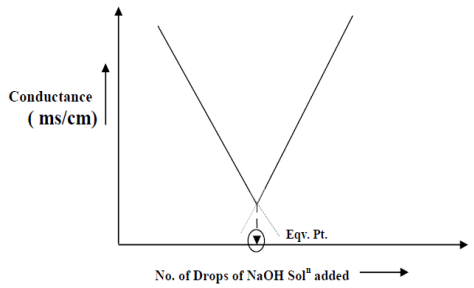
The conductivity cell is inserted into the acid solution in the beaker in such a way that the two electrodes completely dipped into the solution .The cell are connected to the digital conductometer to measure the conductance of the solution. The initial conductance of HCl solution is noted and then NaOH solution is added drop wise from the burette, 10 drops at a time in the beginning, 4 drops at a time near the end point. Near the end point there is a sharp rise in conductance. Beyond the end point 6 to 7 more reading by adding 10 drops of NaOH solution at a time is taken. After each addition of NaOH solution the beaker was shaken gently, waited for a minute and conductance was noted. The conductance against corresponding no. of drops of NaOH solution added are given in Table1.

CONDUCTOMETRIC TITRATION:

S. No	Volume of HCl (mL)	Volume of NaOH (mL)	Measured Conductance (ms/cm)	Corrected conductance $C.C = \left(\frac{V+v}{V} \times C\right)$
1	20	0		
2	20	0.2		
3	20	0.4		
4	20	0.6		
5	20	0.8		
6	20	1.0		
7	20	1.2		
8	20	1.4		
9	20	1.6		
10	20	1.8		
11	20	2.0		
12	20	2.2		
13	20	2.4		
14	20	2.6		
15	20	2.8		
16	20	3.0		
17	20	3.2		
18	20	3.4		

Graph:

Conductance against corresponding no. of drops of NaOH solution added are plotted in graph. It shows the conductometric titrations curve of HCl solution by NaOH solution.



Calculation from graph:

Let the point of intersection of the straight line obtained by plotting conductance against no of drops of NaOH be A drops.

Thus no of drops of NaOH required = (V_1) ml

We know N1V1=N2V2

Strength of NaOH (N1)= N

Volume of NaOH (V1)= mL

Strength of HCl (N2) = ?

Volume of HCl (V2)= 20mL

N1= N2 V2/V1= N

Strength of HCl = N3*Atomic Weight of HCl

= gms

The concentration of HCl solution= gms/Lit.

Discussion: the conductance of the solution measured through conductivity cell. This prior to the experiment the cell constant should be checked and the same cell should be used throughout the experiment. The conductivity of solution depends both the concentration of the electrolyte as well as temperature. The apparatus should thus be cleaned well by distilled water so that no contaminant present in the solution and the conductivity is solely due to HCl and NaOH only and the experiment must be carried out at the same temperature. Addition of NaOH solution, especially near the end point should be taken care of neither there might be some error in finding out the exact equivalence point.

Result:

From the conductometric titration the strength of the supplied HCl solution is found to be gms/Lit..

Weak acid VS strong base

AIM: To determine the strength of the weak acid by titration with strong base by using conduct meter.

APPARATUS: Conductivity Bridge, Conductivity cell, Burette, Beakers, Standard flask, pipette, Burette Stand etc.

CHEMICALS REQUIRED:

Sodium hydroxide, HCl,water,etc….

PRINCIPLE:

At first solution contain $CH_3COO^-$and H^+ ions. Since H^+ ions posses greater mobility it follows that the conductivity is mainly due to H^+ ions. The addition of NaOH is represented by the equation.

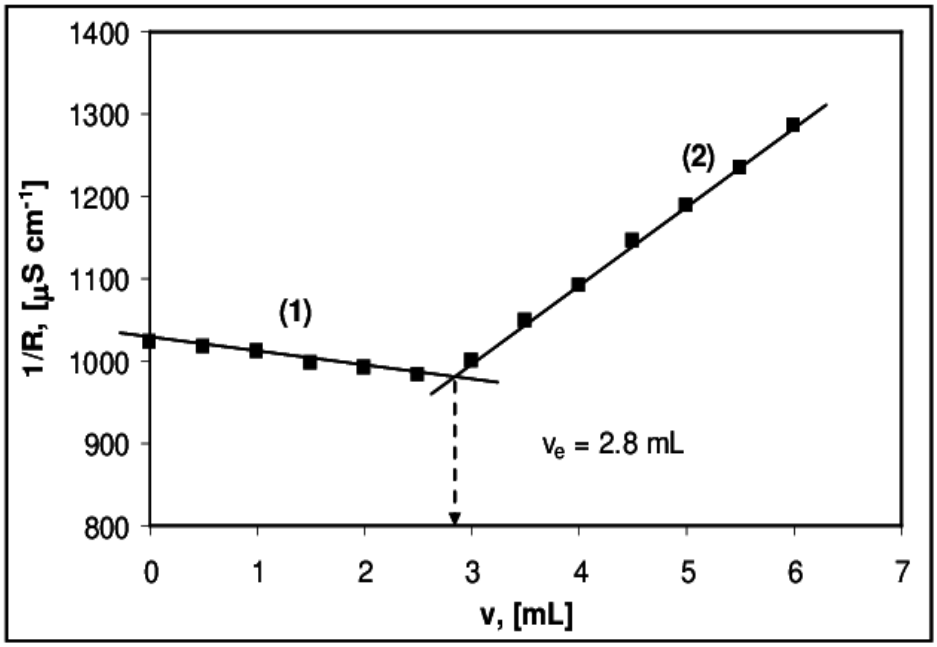
$$H^+ + CH3COO^- + Na^+ + OH^- \longrightarrow CH_3COONa + H_2O$$

As NaOH is added the H^+ ions are removed. The conductivity decreases as Na^+ ions do not process much mobility. As the neutralization point and solutions contains Na^+ ions and Cl^- ions and will have minimum conductance value. If NaOH is further added this will add OH^- ions and so the conductivity increases.

PROCEDURE:

A standard solution of 0.2N NaOH is prepared. Similarly 0.1N CH_3COOH is prepared. 20 ml CH_3COOH of is taken in a 100 ml beaker and to it 20 ml of distilled water is added and kept in a thermostat. The conductivity cell is washed with distilled water and rinsed with acid soln. The cell is kept in acid containing beaker and it is connected to the bridge. The conductivity of the solution is measured by adjusting the reading. NaOH solution is taken into burette and adds 1 ml of solution to acid, stirred well and conductance is measured. Each time 1 ml of base is added to acid stirred well and the conductance is measured. For every instance. Equal numbers of values are taken on either side of the point of maximum. Repeat the procedure of addition of 1 ml NaOH and noting the conductivity of the resulting solution. Take 20-25 readings.

Graph :



S. No	Volume of CH_3COOH (mL)	Volume of NaOH (mL)	Measured Conductance (ms/cm)	Corrected conductance $C.C = \left(\frac{V+v}{V} \times C\right)$
1	20	0		
2	20	0.2		
3	20	0.4		
4	20	0.6		
5	20	0.8		
6	20	1.0		
7	20	1.2		
8	20	1.4		
9	20	1.6		
10	20	1.8		
11	20	2.0		
12	20	2.2		
13	20	2.4		
14	20	2.6		
15	20	2.8		
16	20	3.0		
17	20	3.2		
18	20	3.4		

FORMULA: $N_1V_1 = N_2V_2$

Strength of NaOH (N1)= N

Volume of NaOH (V1)= ml

Strength of CH_3COOH (N2) = ?

Volume of CH_3COOH (V2)= 20mL

N1= N2 V2/V1= N

Strength of CH_3COOH = N3*Atomic Weight of

= =gms/mole

The concentration of CH_3COOHl solution= gms/Lit

RESULT:

The concentration of given unknown solution (**CH_3COOH**) is N

The normality of weak (**CH_3COOH**) determined by titrating against a strong base (NaOH) = gms/Lit.

Experiment-4

Determination of cell constant of a conductivity cell

Aim: - Determination of cell constant of a conductivity cell.

REQUIREMENTS

Apparatus: - Conductometer, Conductivity cell, beaker, Standard flask etc.

Chemicals: - Potassium chloride, distilled water etc.

Theory: - Conductance is the reciprocal of resistance. It is depend upon three factors, number of ions,and nature of ions and mobility of ions towards their respective electrodes. The specific conductanceof electrolyte is decreased by increasing its dilution and equivalent conductance is increased byincreasing its dilution. The observed conductivity of an electrolyte will be equal to its specificconductivity if cell constant is one.

Procedure: - Connect conductivity cell to Conductivity Bridge and keep the cell deepen in distilled water. Satisfactory and reproducible results demand the utmost care and cleanliness in the preparation of solutions and their transfer. Glassware should be cleaned thoroughly with cleansingacid, rinsed repeatedly with tap water, distilled water and finally with conductance water. Do not use organic solvents for drying, use a drying oven.The electrodes of the conductance cell must be immersed in conductance water whenever the cell is not in use. Never touchthe electrodes.All solutions should be prepared using conductance water.In making conductivity measurements, allow the cell to remain in the constant temperature bath for at least fifteenminutes before balancing the bridge for the final reading.

Prepare the solutions of 0.1, 0.2, 0.3, 0.4 and 0.5 N of KCl.

PART I – Determination of cell constant.

Note down the conductance of 0.1, 0.2, 0.3, 0.4, 0.5N and 0.02 N KCl solutions by using conductometer .

Determine the cell constant.

Determine the conductance of all prepared KCl solutions and calculate the specific and equivalentconductance of each solution.Plot a graph Equivalent conductance against concentration of solution.

Formula:

Cell constant = Specific conductance/ observed conductivity

Specific conductance = Cell constant x observed conductivity

Equivalent conductance = K x 1000/C

Part-I

Table-I Measurement of observed conductance for various concentrations

S. No	Conc. of KCl solution in normality	Temparature	Specific Conductance (k)	Observed Conductance (mho)
1	**0.1**	**25**		
2	**0.2**	**26**		
3	**0.3**	**27**		
4	**0.4**	**28**		
5	**0.5**	**29**		
6	**0.02**	**30**		

Calculations:-

1. Specific conductance= Cell constant x observed conductivity
2. Equivalent Conductance= K x 1000/C
3. Cell constant = Specific conductance/ observed conductivity

Result: - The cell constant of the given conductivity cell is = /cm

The Conductance of the given solution = mho.

Expermint-5

POTENTIOMETRY

(Determination of redox potential by emf Titration of Fe^{2+} with $Cr_2O_7^{-2}$)

Aim: to measure the redox potential using potentiometer

REQUIREMENTS

Apparatus: Burette, pipette, volumetric flasks, beakers, magnetic stirrer, potentiometer, SCE, platinum indicator electrode, connecting wires etc.

Chemicals: Potassium dichromate, ammonium iron (II) sulphate and sulphuric acid

Objectives

After performing this experiment you will be able to:

• Discuss how the potential changes with relative concentration ofoxidised/reduced from,

• perform a redox titration of ammonium iron (II) sulphate using potassiumdichromate as oxidizing agent,

• determine the equivalence point of the redox titration by plotting titration curveusing potential change values and amount of oxidizing agent added duringtitration,

• estimate the strength of iron (II) ions in the given solution,

PRINCIPLE

This is an example of redox titration and is based on the oxidation-reduction reactionbetween the titrand and the tirant. Here the end pint is detected using a potentiometer.From the Nernst equation, you know that the potential of a given reaction will dependon the relative concentration of oxidised/reduced from.During their titration, the solution potential changes due to the change in theconcentration of oxidised/reduced form. At one stage, where either of the form is absenti.e. at the end point, there is a sharp change in potential.Potassium dichromate is an oxidising agent and in acid medium; it follows the halfreaction to give Cr (III) as the reduction product.

$$Cr_2O_7^{2-} + 14H + 6e \rightleftharpoons 2\ Cr^{3+} + 7\ H_2O \ldots (1)$$

While Fe^{2+} which is used to titrate $K_2Cr_2O_7$ gets oxidised to Fe^{3+} as per the reaction

$$Fe^{2+} \rightleftharpoons Fe^{3+} + e \ldots (2)$$

The overall ionic equation of this titration can be obtained by adding the above two:

$$Cr_2O_7^{2-} + 6\ Fe^{2+} + 14H^+ \rightarrow 6\ Fe^{3+} + 2Cr^{3+} + 7\ H_2O \ldots (3)$$

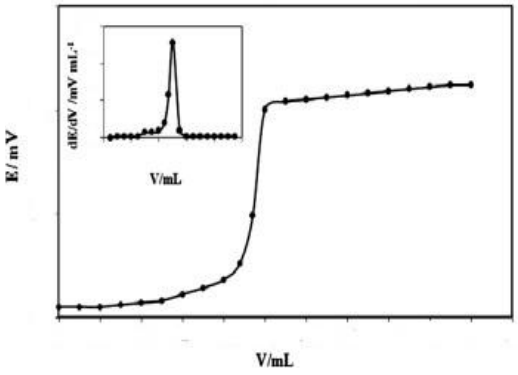
PROCEDURE

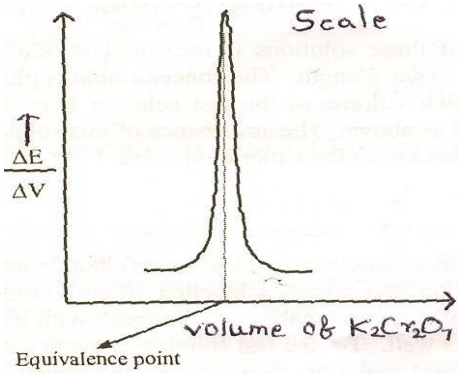
1. Using 100 cm3 volumetric flasks prepare of 0.02 M potassium dichromate solution and 0.10 M ammonium iron (II) sulphate solution. You may have to add sufficient amount of dilute acid to prepare ammonium iron (II) sulphate solution.
2. Take 25 cm3 of given Fe^{2+} solution and add 25 cm3 dilute H_2SO_4 acid and 50 cm3 of distilled water in a 250 cm3 beaker.
3. SCE is used as the Reference electrode. Platinum metal foil, dipped in Fe2+solution is used as the indicator electrode.
4. Standardise the potentiometer using a standard cell before replacing it with the working cell.
5. Add 2 cm3 of 0.02 M $K_2Cr_2O_7$ solution from burette, operate the magnetic stirrer for 2 minutes, stop it, wait it for 1 minute and measure the E.M.F.
6. Repeat the above step, each time adding two more cm3 of $K_2Cr_2O_7$ at a time and go on noting the E.M.F. after each addition.
7. When the volume reached near about 1 cm3 of the expected equivalence point (approximate), add the solution from burette in 0.5 cm3 instalments and note the potential each time.
8. Continue adding these instalments even after the equivalence point (This can be easily observed from the change in measured potential). The change becomes very small. Continue for another 5-6 additions. Note the potential readings.
9. Record the observations in the Observation Table 1.Follow the same procedure for plotting the graphs and locate the equivalence point as given in Experiment No. 3.

10. **Observations**

S. No	**Volume (*V*) of K2Cr2O7(Ml) or (cm3)**	**EMF. of (*E*) the cell (millivolts)**	ΔV	ΔE	$\Delta E / \Delta V$	$V + 1/2\ \Delta V$
1	0					
2	1					
3	2					
4	3					
5	4					
6	5					
7	6					
8	7					
9	8					
10	9					
11	10					
12	11					
13	12					
14	13					
15	14					
16	15					
17	16					
18	17					
19	18					
20	19					

From the plotted graph,





the vol. of $K_2Cr_2O_7$ used corresponding to the equivalent point = 10.5mL

Apply the dilution law to calculate concentration of potassium dichromate solution

$$N_1V_1 = N_2V_2$$

Where

Concentration of Fe2+ solution (N1)= ? M/lit

Volume of Fe2+ solution (V1)= mL

Concentration of $K_2Cr_2O_7$ solution (N2) = M/lit

Volume of $K_2Cr_2O_7$ solution (V2) = mL

The concentration of Fe2+ solution = $N_1 = N_2\ V_1/V_2$

= = N

The strength of the given Ferrous Ammonium Sulphate solution =

n1*Equivalent weight of Fe2+ ions

=N1*

= = gm./mole

RESULT: The concentration of Fe^{+2} solution = N

The strength of the given Ferrous Ammonium Sulphate solution= gm./mole.

Experiment-6
Determination of Strength of an acid in Pb-Acid battery

Aim: To determine the strength of acid in Lead-Acid battery

Required

Apparatus: Burette, Conical flask, Beaker, Burette stand, Pipette and Wash bottles

Chemicals required: Acid from Lead acid battery, NaOH, Oxalic acid, Phenolphthalein indicator

Principle:

Strength of acid in Lead-Acid battery, measure of its ability to neutralize with bases to resist change of pH value of acid due to presence of mineral acids like H_2SO_4.

$$H_2SO_4 + NaOH \rightarrow Na_2SO_4 + H_2O$$

Procedure:

Standardization of Sodium Hydroxide:

Take 20 ml of oxalic acid solution in to the conical flask and add 2 to 3 drops of Phenolphthalein indicator, resulting the complete solution is colorless. The solution is titrated against with Sodium hydroxide solution. The sample solution changes colorless to pink color. The titration stops and note down the burette value. Then titration should be repeated until getting concrete readings.

Determination of strength of an acid:

Take 20 ml of acid solution from lead acid battery in to the conical flask and add 2 to 3 drops of Phenolphthalein indicator, the complete solution is colorless. The solution is titrated against with sodium hydroxide solution. The sample solution changes colorless to pink color. The titration stops and note down the burette value. Then titration should be repeated until getting concrete readings.

1. **Standardization of Sodium hydroxide**:

S. No	Volume of sample solution	Burette reading(mL)		Volume of oxalic acid(mL)
		Initial	Final	
1	20 ml	0		
2	20 ml			

Calculations:

$N_1V_1 = N_2V_2$

Concentration of Sodium Hydroxide (N_1) = ?
Volume of sodium hydroxide (V_1) = ml
Concentration of Oxalic acid (N_2) = 0.1N
Volume of Oxalic Acid (V_2) = ml

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

Concentration of Sodium hydroxide solution N_1= = N

2. Strength of acid in Lead-Acid battery

S. No	Volume of sample solution	Burette reading(mL)		Volume of Sodium Hydroxide(mL)
		Initial	Final	
1	20 ml	0		
2	20 ml			

Calculations:

$N_1V_1 = N_2V_2$

Concentration of Sodium Hydroxide (N_1) = 0.102N
Volume of sodium hydroxide (V_1) = ml
Concentration of Sulphuric acid (N_2) = ?
Volume of Sulphuric acid (V_2) = ml

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

= = N

Concentration of acid in Lead-acid battery N_2= N

The strength of the given acid in Lead –Acid battery = Equivalent weight of H2SO4 xN_2

= = gr/mole

Result: The strength of acid in Lead –Acid battery is **gr/mo**l

Expermint-7

Preparation a Bakelite and Measurement of its mechanical properties

Aim: To prepare phenol-formaldehyde resin.

Required Apparatus: Beakers, conical flask, glass rod, measuring cylinders, fractionation weight box etc.

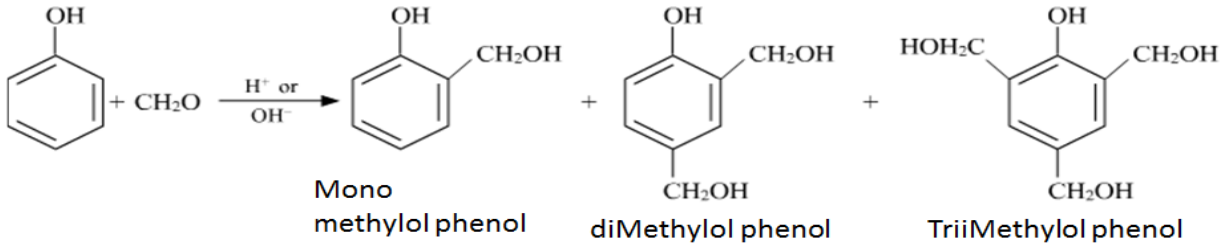
Chemicals: glacial acetic acid,40% formaldehyde solution, phenol, conc. HCl, distilled water.

Theory: phenol resins are condensation polymerization product of phenolic derivative with aldehyde (like formaldehyde). It is prepared by condensing phenol with formaldehyde in the presence of acid or alkaline catalyst.

Reaction:

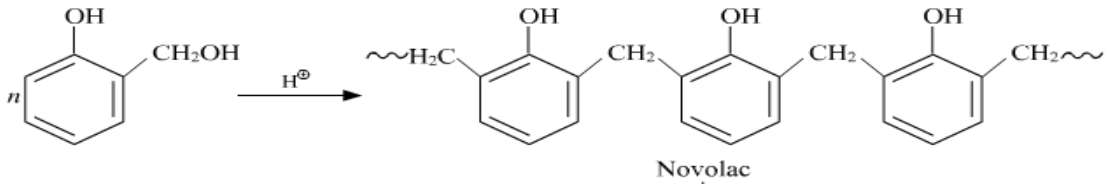
Step-1

The first step is reaction between phenol and formaldehyde, forms mono, di and tri-methylol phenols.



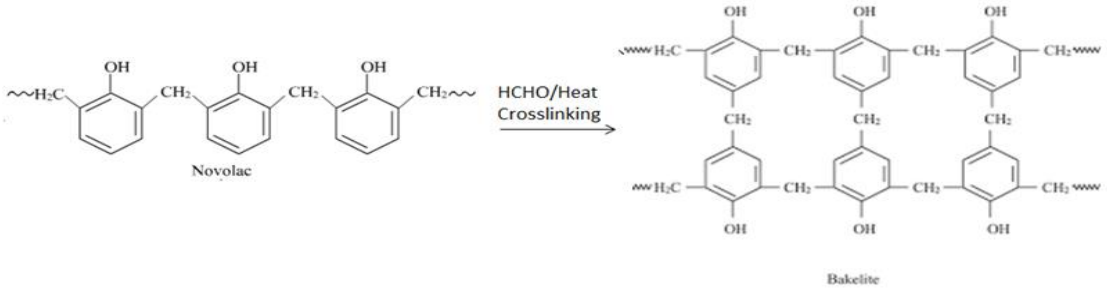
Step-II

When mehtylol phenols are heated with excess of phenol in presence of acid catalyst, the methylol phenols condense with phenol through methylene linkages to from novolacs.



Step-III

Further heating methylol phenol and phenol (or) Resole and Phenol both in the presence of a curing agent (hexamethylene tetramine) products hard, regid, infusible, cross linked polymer is called bakelite.



Further heating of novolac in the presence of hexamethylene tetramine produces hard, rigid, infusible polymer called Bakelite.

Procedure:

1. Place 5ml of glacial acetic acid and 2.5ml of formaldehyde solution in 500ml beaker.
2. Add 2gm of phenol and 1ml of conc.HCl solution in it.
3. Heat the solution slowly with constant stirring for 5mins.
4. A large mass of pink plastic is formed.
5. The residue obtained is washed several times with distilled water.
6. Dry the product and calculate the yield accurately.

Properties:

1. It is insoluble in almost all organic solvents
2. It is water –resistant.
3. It has good electrical insulating character.
4. It is thermally stable.
5. It is highly resistant to non-oxidizing acids, salts and many organic s
6. It is attacked by alkalis because of presence of –OH groups.

Uses:

1. For making telephone parts, radio and T.V. cabinets
2. For making electrical equipment like switches, plugs, holders, switch boards, heaters handles, etc.
3. It is used in the preparation of paints and varnishes and protective coatings
4. In the production of ion–exchange resins

Result: The weight of obtained Bakelite is **gms.**

Experiment-8

Verify lambert-Beer's law

AIM: To verify Lambert –beer 's law for $KMnO_4$colorimetrically.

REQUIREMENTS:

Apparatus: Colorimeter cuvette, five 20 × 150 mm test tubes, two 10 mL pipets or graduated cylinders two 100 ml beakers test tube rack, stirring rod and tissues (preferably lint-free).

Chemicals: 0.01M $KMnO_4$ solution distilled water

THEORY:

The primary objective of this experiment is to determine the concentration of an unknown KMnO4 solution. The $KMnO_4$ solution used in this experiment has a blue color, so Colorimeter users will be instructed to use the red LED. A higher concentration of the colored solution absorbs more light (and transmits less) than a solution of lower concentration.

We should prepare five of known concentration (standard solutions). Each solution is transferred to a small, rectangular cuvette that is placed into the Colorimeter or Spectrometer. The amount of light that presence of the solution and strikes the photocell is used to compute the absorbance of each solution. When graph absorbance *vs*. concentration for the standard solutions, a direct relationship should result. The direct relationship between absorbance and concentration for a solution is known s *Beer's law*.

You will determine the concentrtion of unknown $KMnO_4$solution by measuring its absorbance. By locating the absorbance of the unknown on the vertical axis of the graph, the corresponding concentration can be found on the horizontal axis. The concentration of the unknown can a so be found using the slope of the Beer's law curve.

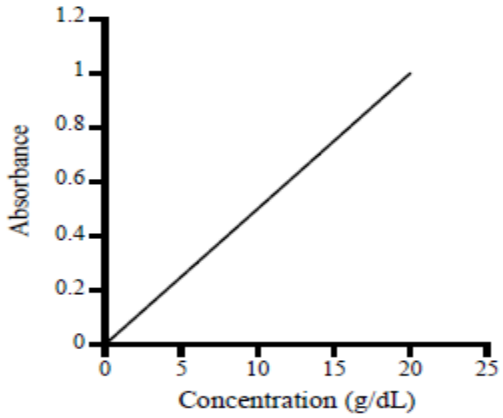


Figure: Beers law graph Absorbance Vs Concentration.

PROCEDURE:

Obtain small volumes o0. 01M $KMnO_4$ solution and distilled water in separate beakers. Label five clean, dry, test tubes 1–5. Use pipets to prepare five standard solutions according to the chart below. Thoroughly mix each solution with a stirring rod. Clean and dry the stirring rod between uses.

Table-1: Preparation of Standard $KMnO_4$ Solution

Test tube	$KMnO_4$ (0.01) ml	Distilled H_2O (ml)	Concentration (M)
1	2.0 ml	8.0 ml	0.002
2	4.0 ml	6.0 ml	0.004
3	6.0 ml	4.0 ml	0.006
4	8.0 ml	2.0 ml	0.008
5	10.0 ml	0 ml	0.01
Unknown	6.0ml	4.2ml	0.008

Prepare a *blank* by filling a cuvette 3/4 full with distilled water. To correctly use cuvettes, remember

- Wipe the outside of each cuvette with a lint-free tissue.
- Handle cuvettes only by the top edge of the ribbed sides.
- Dislodge any bubbles by gently tapping the cuvette on a hard surface.
- Always position the cuvette so the light passes through the clear sides.

You are now ready to collect absorbance-concentration data for the five standard solutions.

- Using the solution in Test Tube 1, rinse the cuvette twice with ~1 mL amounts and then fill it 3/4 full. Wipe the outside with a tissue and place it in the device (Colorimeter or Spectrometer). Close the lid on the Colorimeter.
- When the absorbance readings stabilize,
- Discard the cuvette contents as directed. Using the solution in Test Tube 2, rinse and fill the cuvette 3/4 full. Wipe the outside and place the cuvette in the device (close the lid of the Colorimeter). When absorbance readings stabilize,
- Repeat the procedure for Test Tubes 3 and 4. Trial 5 is the original 0.01M $KMnO_4$solution.

Note: Do not test the unknown solution until Step 9.

Determine the absorbance value of the unknown $KMnO_4$ solution.

- Obtain about 5 mL of the *unknown* $KMnO_4$ in another clean, dry, test tube. Record the number of the unknown in your data table.
- Rinse the cuvette twice with the unknown solution and fill it about 3/4 full. Wipe the outside of the cuvette, place it into the device. (Close the lid of the Colorimeter.)
- Read the absorbance value displayed in the meter. When the displayed absorbance value stabilizes, record its value as Trial 6 in your data table.
- Select Interpolate from the Analyze menu. Find the absorbance value that is closest to the absorbance reading you obtained in Step c above. Determine the concentration of your unknown $KMnO_4$ solution and record the concentration in your data table.
- Dispose of any of the remaining solutions as directed.

Absorbance of the solution:

Trial	Concentration (moL/L)	Absorbance
1		
2		
3		
4		
5		
6	**Unknown solution**	

Calculations:

From graph we know the concentration of unknown $KMnO_4$ solution M/Lit

Amount of unknown KMnO4 solution= **(moL/L)**

$$\text{Amount of unknown solution} = \frac{\text{Conc of Un known solution} \times \text{At. wt of Mn}(54.94) \times 100}{1000}$$

= = **(mol/l)**

Report: the given unknown solution verifying beers and lamberts law**.**

1. Concentration of unknown solution = **(mol/l**M/Lit
2. Amount of Unknown solution = **mg**/Lit (or) ppm

Experiment-9
THIN LAYER CHROMATOGRAPHY
(Separating Food Colors Using Thin Layer Chromatography)

Aim: Thin layer chromatography to separate commercial food colors and achieve the following objectives:

1- To practice the technique of thin layer chromatography and be familiar with the different scientific terms used.

2- To identify which commercial food color is a mixture of dyes and which one contains only a single dye.

3- To check if any single dye is found in more than one commercial food color.

Required

Glassware and Tools:

Thin layer chromatography, Capillary tube to spot samples, Beaker, tall-form, 500-mL, watch glass, large (to fit beaker), Scissors, Pencil, Ruler.

Chemicals and solutions: Commercial food colors, Sodium chloride solution, NaCl, 0.1% (w/v)

Safety precautions:

- Wear goggles and gloves particularly when handling the chromatography paper and colors.
- The dyes in the food colors are slightly hazardous by ingestion, inhalation, eye and skin contact. Some may be absorbed through skin and some may be a skin contact sensitizer. All are irritating to skin and eyes. Avoid contact with eyes, skin, and clothing.

Principle of Thin layer chromatography:

The principle of separation is adsorption. one or more compounds are spotted in the layer of adsorbent coated on a chromatographic plate.The mobile phase solvent flows through because of capillary action (against to gravitational force)The compounds move according to their affinities towards the adsorbent. The compounds with more affinity towardsthe stationary phase traveler slower. The compounds with Less affinity towardsthe stationary phase traveler slower.Thus the components are separated on a thin layer chromatographic plate based on the affinity of the components towards the stationary phase.

Procedure:

1. Cut a 11 cm × 8 cm piece of thin layer plate. Handle the paper carefully by the edges to avoid compacting or contaminating the analysis area.
2. Orientate the thin layer chromatography paper so that it is 8 cm wide and 11 cm tall.

3. Using a ruler and **pencil** (**not a pen**), draw a faint line 1.5 cm from the bottom across the entire width of the plate.
4. Using the same ruler and pencil, draw three small dots. Measure **2 cm** from one edge for the first dot and then add a dot every 2 cm across the line. Label the dots using numbers from 1 to 3.
5. Obtain the commercial food color samples.
6. Using a clean capillary for each dye sample, spot on the thin layer chromatography paper by putting the capillary into the dye sample solution and then touching the tip of the capillary gently onto a pencil dot. After the first dot is **dry**, repeat the procedure to increase the concentration of the sample BUT do not increase the size of the spot.
7. Wait about 3 minutes for the samples on the thin layer plate to completely dry (you may use air dryer or an oven at 80°C).
8. While the sample is drying obtain a tall-form beaker and a watch glass that will fit over the container.
9. Pour some of the 0.1% NaCl solution enough to form a level of about 1 cm into the beaker and **cover the top with a watch glass**. This is the chromatography chamber. The 0.1% NaCl is the developing solvent. Avoid adding the solution to higher level in the beaker.
10. Remove the watch glass from the beaker and carefully place the thin layer plate cylinder into the chamber with the sample end down. Do not get any solvent on the upper portion of the thin layer plate. . The sample spots must remain above the level of the solvent. If the solvent level is too high, the samples will dissolve into the solvent and washed off.
11. Place the watch glass back on the beaker and allow the chromatogram to develop 15–20 minutes until the solvent is within 1–2 cm of the top of the plate, then remove the plate from the beaker and lay it flat.
12. With a pencil, immediately draw a line to mark the distance the solvent traveled to the top of the chromatography plate. This is called the **solvent front**.
13. Measure the distance from the pencil line at the bottom of the chromatography plate to the solvent front. Record this distance in cm.
14. In pencil, trace the shape of each dye band or spot to mark the location of each separated band. This should be done immediately because the color and brightness of some spots may fade over time.
15. Measure the distance traveled in cm by each dye from the line at the bottom of the plate to the center of each band. Calculate *Rf* Value for each dye.

$$\text{Retardation factor Rf} = \frac{\text{Distance traveled by the solute}}{\text{Distance travelled by the solvent front}}$$

Table-1: Visual identification of commercial color components

Spot number	Commercial color Name	Number of bands separated	Observed colors of bands
1			
2			
3			
4			

Table-2: Identification of separated components by Rf values

Spot Number	Commerical color Name	Distance Solvent traveled	Distance Dye traveled	Rf
1				
2				
3				
4				

Results:using thin layer chromatography food colors are separated.

Experiment-10

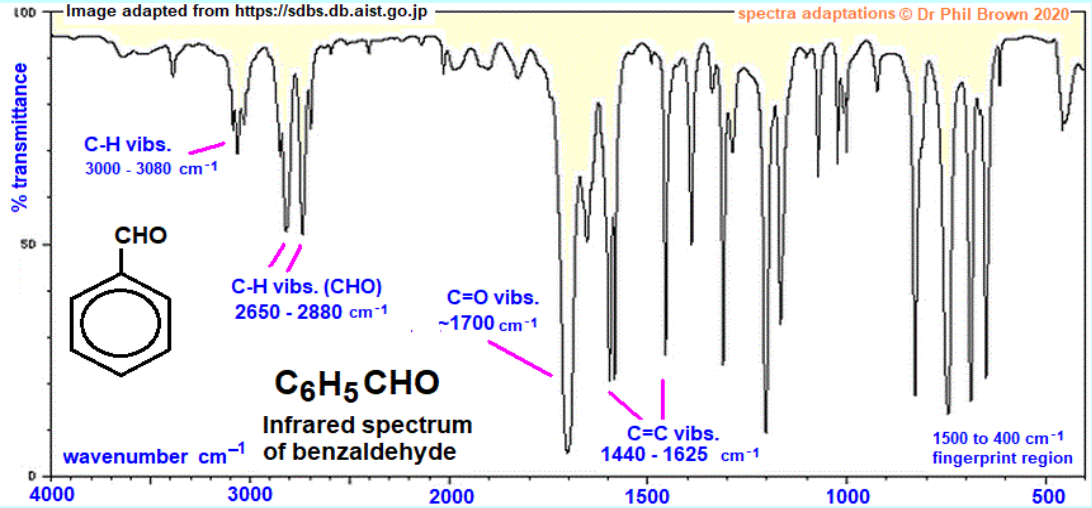
IDENTIFICATION OF SIMPLE ORGANIC COMPOUNDS BY IR

AIM :- Identification of simple Organic Compounds by IR

Example :- 1. **Infrared spectrum of benzaldehyde.**

Interpreting the infrared spectrum of benzaldehyde

molecular spectroscopy analyzing infrared spectra of benzaldehyde



ANALYSIS :-

Spectra obtained from a liquid film of benzaldehyde. The right-hand part of the of the infrared spectrum of benzaldehyde wavenumbers ~1500 to 400 cm^{-1} is considered the fingerprint region for the identification ofbenzaldehyde and mostorganic compounds. It is due to a unique set of complex overlapping vibrations of the atoms of the molecule of benzaldehyde.

Interpretation of the infrared spectrum of benzaldehyde

The most prominent infrared absorption lines of benzaldehyde-

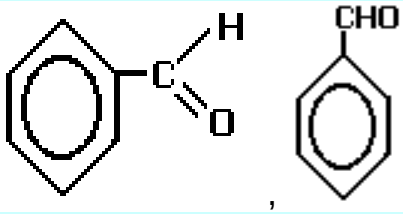
At wavenumbers 3000 to 3080 cm^{-1} you get the C-H vibrations from the benzene ring.

At wavenumbers 2650 to 2880 cm^{-1} you get the C-H vibration absorptions from the aldehyde group.

At ~1700 cm^{-1} you get the characteristic C=O stretching vibrations of the carbonyl group.

Between wavenumbers 1440 and 1625 cm-1 you get several absorption bands between due to C=C vibrations in the benzene ring.

The absence of other specific functional group bands will show that particular functional group is absent from the benzaldehyde molecular structure.

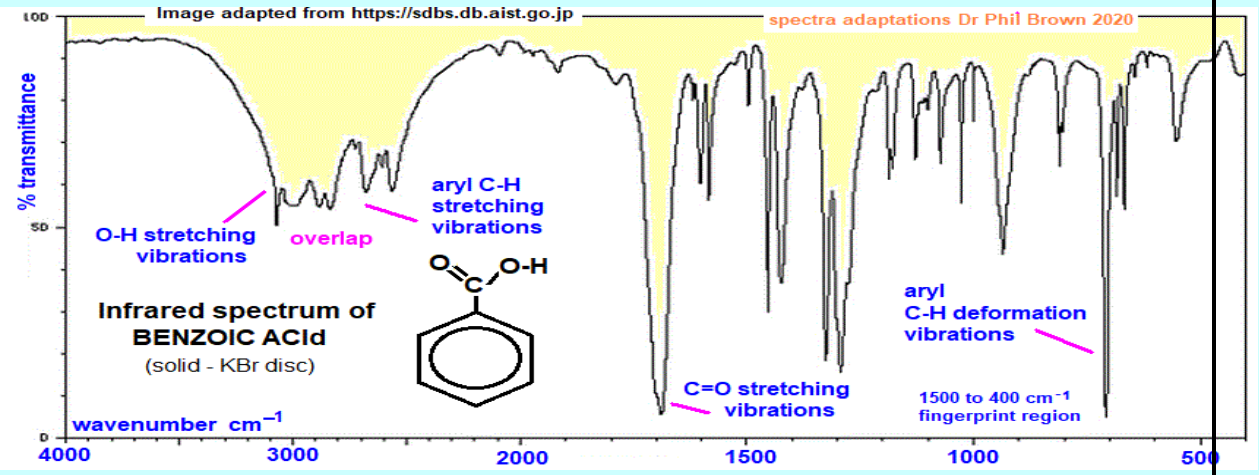
benzaldehyde C_7H_6O, C_6H_5CHO , 

,

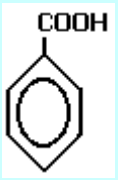
Example :- 2. Infrared spectrum of Benzoic acid

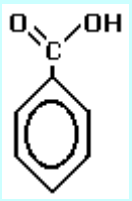
Interpreting the infrared spectrum of benzoic acid

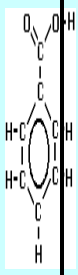
Doc molecular spectroscopy analysing infrared spectra of benzoic acid



Spectra obtained from a liquid film of benzoic acid. The right-hand part of the of the infrared spectrum of benzoic acid, wavenumbers ~1500 to 400 cm^{-1} is considered the fingerprint region for the identification of benzoic acid and most organic compounds. It is due to a **unique** set of complex overlapping vibrations of the atoms of the molecule of benzoic acid.

Benzoic acid (benene - carboxylic acid), **$C_7H_6O_2$**, **C_6H_5COOH** , 

, 

, 

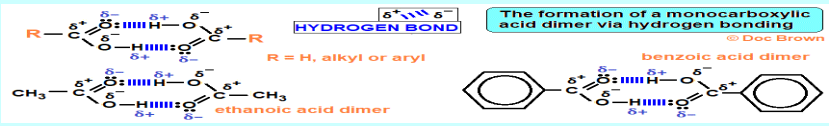
Interpretation of the infrared spectrum of benzoic acid

The most prominent infrared absorption lines of benzoic acid

A most characteristic absorption band is the broad **O-H stretching vibration** bands at wavenumbers ~3300 to 2500 cm^{-1}, the breadth is caused by interference of hydrogen bonding interactions, common to all hydrogen bonded molecules with a hydroxyl group.

The intra molecular hydrogen bond **C_6H_5-C=$O^{\delta-}$-$H^{\delta+}$:$O^{\delta-}$-O-C_6H_5** in benzoic acid.

The stretching O-H vibrations overlap the C-H aryl stretching vibrations at wavenumbers 3080 to 3030 cm^{-1}.



The 2nd characteristic band is the absorption due to the **carbonyl (C=O) stretching vibrations** wavenumbers 1700 to 1680 cm^{-1} for aryl carboxylic acids like benzoic acid.

There are **aryl C-H (C=C-H) stretching vibration** bands at wavenumbers ~1100 to 1000 cm^{-1} (for monosubstitution benzene compounds).

There are quite prominent **aryl C-H deformation vibration** absorptions at wavenumbers 770 to 690 cm^{-1}, you can see strong absorption at a little over wavenumber 700 cm^{-1} (for monosubstitution benzene compounds).

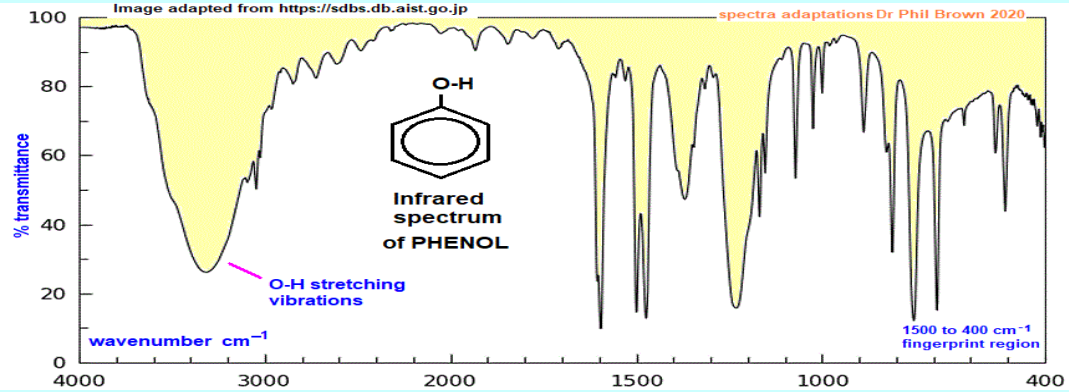
The presence of strong absorption at wavenumbers for C=O and O-H stretching vibrations are very indicative of a carboxylic acid group in the benzoic acid molecule, but **not proof or exclusive** e.g. you can have a hydroxy-ketone!

The absence of other specific functional group bands will show that a particular functional group is absent from the benzoic acid molecular structure.

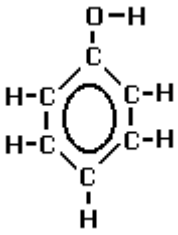
Example :- 3. Infrared spectrum of phenol

Interpreting the infrared spectrum of phenol

molecular spectroscopy analyzing infrared spectra of phenol



Spectra obtained from a liquid film of phenol. The right-hand part of the of the infrared spectrum of phenol, wavenumbers ~1500 to 400 cm^{-1} is considered the fingerprint region for the identification of phenol and most organic compounds. It is due to a **unique** set of complex overlapping vibrations of the atoms of the molecule of phenol.

Phenol, C_6H_6O, C_6H_5OH,  , 

Interpretation of the infrared spectrum of phenol

The most prominent infrared absorption lines of phenol

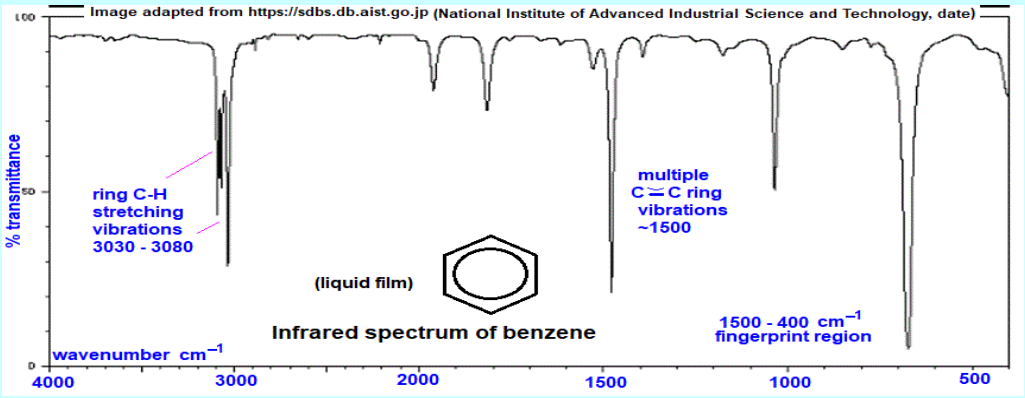
The most characteristic absorption band is the broad O-H stretching vibration bands at wavenumbers ~3550 to 3230 cm^{-1}, the breadth is caused by interference of hydrogen bonding interactions, common to all hydrogen bonded molecules with a hydroxyl group.

The intermolecular hydrogen bond $C_6H_5\text{-}O^{\delta-}\text{-}H^{\delta+}\|\|\|\|\|:O^{\delta-}\text{-}C_6H_5$ in phenol.

Example :-4. Infrared spectrum of Benzene

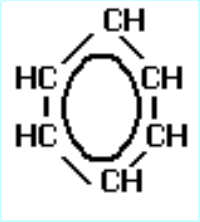
Interpreting the infrared spectrum of benzene - C_6H_6

molecular spectroscopy analysing infrared spectra



Spectra obtained from a liquid film of benzene. The right-hand part of the of the infrared spectrum of benzene,wavenumbers ~1500 to 400 cm^{-1} is considered the fingerprint region for the identification of benzene andmost organic compounds. It is due to a **unique** set of complex overlapping vibrations of the atoms of themolecule of benzene.

benzene C_6H_6, skeletal formula  **,**

structural/displayed formula 

Interpretation of the spectrum of benzene C_6H_6

The most prominent infrared absorption lines of benzene

There are characteristic C-H stretching vibrations ~3030 to 3080 cm^{-1}, and

characteristic C-H stretching vibrations ~3030 to 3080 cm^{-1},

and characteristic C=C benzene ring vibrations.

The absence of other specific functional group bands will show that

particular functional group is absent from the benzene molecular structure.

EXPERMENT - 11

PREPARATION OF NANOMATERIALS

Aim:
To prepare the Magnetite Nanoparticles.

Apparatus:
100 ml Glass Beaker, 5ml Measuring jar, *50*ml Burette, Magnet, Plastic weigh boat, Wash bottle, Glass rod, Electronic balance etc.

Chemical
lM FeCl3, 2M FeCl2, lM NH_3, 25% Tetraethyl ammonium hydroxide.

Procedure

In a 100 ml Glass beaker, add *5* ml of lM FeCl3 and 1 ml of 2M FeCl2 solution, place a magnet under the container and stir continuously add about 1 ml of 1 M NH3 up to *50* ml every 10 seconds over a period of 5-10 minutes (**Do not add the solution all at once**). Then Black particles begin to form and solid particles will settle down followed by transfer and remove the clear solution without losing the solid.

Now transfer the solid to a plastic weigh boat with the help of a small amount of water from a wash bottle. Use the magnet to keep the solid at the bottom of the plastic weigh boat and remove the liquid. Rinse the solid once more with water and transfer. Now remove the magnet and add 1-2 ml of 25% Tetraethyl Ammonium hydroxide with continuously stirring by a glass rod for 1-2 minutes. Then ferrofluid will form. Use a strong magnet to attract the ferrofluid to the bottom of the weighing boat Pour off and discard the dark liquid. Move the magnet around and again pour off any liquid. If the ferrofluid does not spike, continue to move the strong magnet around, pouring off any liquid. Examine the spikes that form as you move the magnent around finally collect the obtained **spikes of Magnetite Nanoparticles** and calculate the weight of Magnetite Nanoparticle by using electronic balance.

Result

The weight of obtained Magnetite Nanoparticles = gm

EXPERMENT- 12

Estimation of Ferrous Iron by Dichrometry

Aim: To estimate the amount of Ferrous iron in the whole of the given solution using a standard solution of Potassium Dichromate.

Required

Apparatus: 100 ml standard flask, Burette,250 ml Conical Flask,20 ml Pipette, Funnel & Simple balance with Fractional weights.

Chemicals: Potassium dichromate ($K_2Cr_2O_7$),Sulphuric acid (H_2SO_4), Syrupy phosphoric acid (H_3PO_4), Diphenylamine, Ferrous iron solution & distilled water.

Principle: Ferrous Iron is oxidized to Ferric iron by Potassium Dichromate in acid solution. The completion of oxidation reaction is marked by the appearance of Blue violet color of Diphenylamine, which is used as an internal indicator.

Chemical reaction:

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

Only one electron is necessary to reduce Fe(III) to Fe(II)

$$Fe^{3+} + e^- \rightarrow Fe^{2+}$$

Therefore, 1 mole of $Cr_2O_7^{2-}$(the oxidizing agent) reacts with 6 moles of Fe^{2+} (the reducing agent) to form 6 moles of Fe^{3+} and 2 moles of Cr^{3+}. Thus, in net ionic form:

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

The molecular form ofthe reaction equation can be written as:

$$K_2Cr_2O_7 + 6Fe(NH_4)_2(SO_4)_2 + 7H_2SO_4 \rightarrow 3Fe_2(SO_4)_3 + Cr_2(SO_4)_3 + K_2SO_4 + 6(NH_4)_2SO_4 + 7H_2O$$

The 1:6 mole ratio with respect to the amounts of $Cr_2O_7^{2-}$ and Fe^{2+} consumed will provide the stoichiometric basis for all of the calculations in this experiment.

Procedure:

Preparation of standard potassium dichromate: Weigh out accurately the given pure crystalline sample of potassium dichromate and transfer into 100 ml standard (volumetric) flask provided with a funnel. Dissolve the dichromate in a small quantity of distilled water, and make upto the mark. The contents in the flask are shaken well for uniform concentration. Calculate the normality of potassium dichromate.

Preparation of Standard (K_2Cr_2O) solution:

W1 = Weight of bottle + substance($K_2Cr_2O_7$) = gms

W2 = Weight of bottle = gms

Weight of substance ($K_2Cr_2O_7$) = (W1-W2) = gms.

Normality of the $K_2Cr_2O_7$solution = ((W1-W2) X 10)/Equivalent Weight

N = ((W1-W2) X 10)/49 = N

Estimation of Iron: Make up given solution upto the mark with distilled water and shake the flask for uniform concentration. Rinse the pipette with the ferrous solution and pipette out 20ml into a clean conical flask add 10ml of the acid mixture (sulphuric acid and phosphoric acid), and four to five drop of diphenylamine indicator. Fill the burette with the prepared potassium dichromate solution after rinsing it, with the same. Titrate the solution in the conical flask against the standard potassium dichromate from the burette till the color changes to blue violet. Repeat the titrations for concurrent titer values.

Table-1: Estimation of Iron

S.No	Volume of standard iron solution	Burette readings(mL)		Final Volume of $K_2Cr_2O_7$ Consume (mL)
		Initial	Final	
1	20.0ml	0		
2	20.0ml			

Calculation:

N1 = Normality of $K_2Cr_2O_7$ solution = N

N2 = Normality of Ferrous iron solution = ?

V1 = Volume of $K_2Cr_2O_7$ solution = ml

V2 = Volume of Ferrous iron solution = 20 ml

$$N1\ V1 = N2\ V2$$

$$N2 = (N1\ V1)/V2$$

Normality of Ferrous iron solution = N2 = (N1 V1)/V2

= = N

Amount of Ferrous iron present in the whole of the given solution (100 ml) =

= **(N2 X 55.85X100)/1000**

= X55.85X100/1000

= mg/l

Result: Amount of Ferrous iron present in the whole of the given solution (100 ml) = **mg/l.**