DRONACHARYA
College of Engineering

LABORATORY MANUAL

B.Tech. Semester- I/ II

CHEMISTRY LAB
Subject code: BSC-101P

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DEPARTMENT OF APPLIED SCIENCE & HUMANITIES
DRONACHARYA COLLEGE OF ENGINEERING
KHENTAWAS, FARRUKH NAGAR, GURUGRAM (HARYANA)

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Vision and Mission of the Institute

Vision:

"To impart Quality Education, to give an enviable growth to seekers of learning, to groom them as World Class Engineers and managers competent to match the expending expectations of the Corporate World has been ever enlarging vision extending to new horizons of Dronacharya College of Engineering."

Mission:

M1. To prepare students for full and ethical participation in a diverse society and encourage lifelong learning by following the principle of 'Shiksha evam Sahayata' i.e. Education & Help.

M2. To impart high-quality education, knowledge and technology through rigorous academic programs, cutting-edge research, & Industry collaborations, with a focus on producing engineers& managers who are socially responsible, globally aware, & equipped to address complex challenges.

M3. Educate students in the best practices of the field as well as integrate the latest research into the academics.

M4. Provide quality learning experiences through effective classroom practices, innovative teaching practices and opportunities for meaningful interactions between students and faculty.

M5. To devise and implement program of education in technology that are relevant to the changing needs of society, in terms of breadth of diversity and depth of specialization.

Vision and Mission of the Department

Vision

To lay a strong foundation for the first year students of the engineering discipline in the area of Applied Sciences and Humanities with a view to make them capable of innovating and inventing engineering solutions and also develop students as capable and responsible citizens of our nation.

Mission

- To build strong fundamental knowledge and ability for application in students and make them capable to apply knowledge of mathematics and science to the solution of complex engineering problems.
- To impart knowledge, leading to understanding between engineering and other core areas of Applied Sciences and Humanities.
- To provide students the basic tools of analysis, as well as the knowledge of the principles on which engineering is based.
- To strive to inculcate the scientific temper and the spirit of enquiry in the students.
- To make students achieve a superior level in communication and presentation skills.
- To foster values and ethics and make students responsible citizens of India.
- To strive to inculcate the scientific temper and the spirit of enquiry in the students.
- To pursue inter-disciplinary research for the larger good of the society

Program Educational Objectives (PEOs)

PEO1: To provide students with a sound knowledge of mathematical, scientific and engineering fundamentals required to solve real world problems.

PEO2: To develop research oriented analytical ability among students and to prepare them for making technical contribution to the society.

PEO3: To develop in students the ability to apply state-of-the–art tools and techniques fordesigning software products to meet the needs of Industry with due consideration for environment friendly and sustainable development.

PEO4: To prepare students with effective communication skills, professional ethics and managerial skills.

PEO5: To prepare students with the ability to upgrade their skills and knowledge for life-long learning.

Program Outcomes (POs)

PO1: Engineering knowledge: Apply the knowledge of mathematics, science, engineering fundamentals, and an engineering specialization to the solution of complex engineering problems.

PO2: 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.

PO3: 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.

PO4: 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.

PO5: 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.

PO6: 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.

PO7: 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.

PO8: Ethics: Apply ethical principles and commit to professional ethics and responsibilities and norms of the engineering practice.

PO9: Individual and team work: Function effectively as an individual, and as a member or leader in diverse teams, and in multidisciplinary settings.

PO10: 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.

PO11: 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.

PO12: 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.

Program Specific Outcomes (PSOs)

PSO1: Analyze, identify and clearly define a problem related to chemistry solving user needs by selecting, creating and evaluating.

PSO2: Design, implement and evaluate processes, components and/or programs using modern techniques, skills and analytical methods.

PSO3: Develop effective solutions by using research based knowledge and research methods in the fields of chemical analysis, sustainable development and environmental protection.

University Syllabus

Course Objective: The chemistry laboratory course will consist of experiments illustrating the principles of chemistry relevant to the study of science and engineering.

LIST OF EXPERIMENTS: -

1. Determination of surface tension of given liquid by drop number method.
2. Determine the viscosity of given liquid by using Ostwald's viscometer / Redwood viscometer.
3. Calculate the Rf value of given sample using Thin layer chromatography / Paper chromatography.
4. Removal of Ca2+ and Mg2+ hardness from given water sample using ion exchange column.
5. Determination of chloride content in given water sample.
6. Calculate the strength of strong acid by titrating it with strong base using conductometer.
7. Calculate the emf value of given cell.
8. To prepare the of urea formaldehyde and phenol formaldehyde resin.
9. To determine the rate constant of a reaction.
10. To Prepare iodoform.
11. Calculate the saponification value / acid value of given oil sample.
12. Chemical analysis of two anions and two cations in given sample of salt.
13. Determination of the partition coefficient of a substance between two immiscible liquids.
14. To determine the total hardness of given water sample by EDTA method.

Note: At least 08 experiments are to be performed by the students.

Course Outcomes

CO1. Students are able to understand concept of hardness.
CO2. Students are able to synthesize polymers and other organic compounds.
CO3. Students are able to measure the properties of different lubricant samples.
CO4. Students are able to apply different analytical techniques .
CO5. Students are able to measure surface tension of different liquids.

CO-PO Mapping

	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12
CO1		1		1		2	3	2	1		1	1
CO2	2	2		1		3	3		1		1	1
CO3	2	1		2		1		2	1			1
CO4	1	2		2		2		1	1		2	2
CO5			2	2				1	2		2	2
CO												

CO-PSO Mapping

	PSO1	PSO2	PSO3
CO1		1	2
CO2		1	2
CO3		2	1
CO4		2	2
CO5		2	2
CO		**2**	**2**

List of Experiments Mapped with CO

S.No.	Name of the Experiment	Course Outcome	Page No.
1	To prepare urea formaldehyde resin	CO2	1-2
2	To prepare phenol formaldehyde resin	CO2	3-5
3	To determine the total hardness of the given water sample by EDTA method	CO1	6-8
4	To determine viscosity of given liquid by using Redwood viscometer	CO3	9-12
5	To prepare iodoform	CO2	13-14
6	To calculate the R_f value of given sample using paper chromatography	CO4	15-18
7	To determine the surface tension of the given liquid by drop number method.	CO5	19-21
8	To determine chloride content in the given water sample	CO4	22-24
9	To calculate the strength of strong acid by titrating it with strong base using conductometer.	CO4	25-27
10	To determine the saponification value of the given oil sample	CO3	28-30

DOs and DON'Ts

DO'S

The Chemistry laboratory must be a safe place in which to work and learn about Chemistry. Most of these involve just using common sense.

1. Use protective clothing all the time (e.g. lab coat and safety glasses).
2. Be familiar with your lab assignment **before** you come to lab. Follow all written and verbal instructions carefully. Observe the safety alerts in the laboratory directions. If you do not understand a direction or part of a procedure, ask the teacher before proceeding.
3. Wash acid, base, or any chemical spill off of yourself immediately with large amounts of water. Notify your teacher of the spill.
4. Clean up spills immediately. If you spill a very reactive substance such as an acid or base, notify the people in the area and then obtain assistance from your teacher. Acid spills should be neutralized with baking soda, base spills with vinegar before cleaning them up.
5. If chemical substances get in your eye, wash the eye out for 15 minutes. Hold youreye open with your fingers while washing it out.
6. Place the reagents in a systemic manner. If you burn yourself on a hot object, immediately hold the burned area under cold water for 15 minutes.
7. Observe good housekeeping practices. Work areas should be kept clean and tidy at all times. Only lab notebooks or lab handouts should be out on the table while performing an experiment. Books and book bags should not be on the lab table. Passageways need to be clear at all times.
8. Always add acid to water and stir the solution while adding the acid. Never addwater to an acid.
9. Report all accidents to your teacher.
10. Thoroughly clean your laboratory work space at the end of the laboratory session.
11. Make sure that all equipment is clean, and returned to its original place.

DON'TS

1. Work in the laboratory without an instructor present. Work only with your labpartner(s). Do not venture to other lab stations for any reason.
2. Wear bulky or dangling clothing.
3. Eat or drink in the laboratory. Don't chew on the end of a pen which was lying onthe lab bench.
4. Use Mobile Phones.
5. Directly touch any chemical with your hands. Never taste materials in the laboratory.
6. Waste the reagents.
7. When entering the lab/classroom, do not touch any equipment, chemicals, or other materials without being instructed to do so. Perform only those experiments authorized by the instructor.
8. When weighing never place chemicals directly on the balance pan. **Never weigh a hot object**.

General Safety Precautions

Precautions (In case of Injury or Electric Shock)

1. To break the victim with live electric source, use an insulator such as fire wood or plastic to break the contact. Do not touch the victim with bare hands to avoid the risk of electrifying yourself.
2. Unplug the risk of faulty equipment. If main circuit breaker is accessible, turn the circuit off.
3. If the victim is unconscious, start resuscitation immediately, use your hands to press the chest in and out to continue breathing function. Use mouth-to-mouth resuscitation if necessary.
4. Immediately call medical emergency and security. Remember! Time is critical; be best.

Precautions (In case of Fire)

1. Turn the equipment off. If power switch is not immediately accessible, take plug off.
2. If fire continues, try to curb the fire, if possible, by using the fire extinguisher or by coveringit with a heavy cloth if possible isolate the burning equipment from the other surrounding equipment.
3. Sound the fire alarm by activating the nearest alarm switch located in the hallway.
4. Call security and emergency department immediately:

Emergency : Reception

Security : Main Gate

Guidelines to students for report preparation

All students are required to maintain a record of the experiments conducted by them. Guidelines for its preparation are as follows: -

1) All files must contain a title page followed by an index page. ***The files will not be signed by the faculty without an entry in the index page.***

2) Student's Name, Roll number and date of conduction of experiment must be written on all pages.

3) For each experiment, the record must contain the following

(i) Aim/Objective of the experiment
(ii) Pre-experiment work (as given by the faculty)
(iii) Lab assignment questions and their solutions
(iv) Test Cases (if applicable to the course)
(v) Results/ output

Note:

1. Students must bring their lab record along with them whenever they come for the lab.

2. Students must ensure that their lab record is regularly evaluated.

3. Students must bring their lab coats, whenever they come for chemistry lab.

Lab Assessment Criteria

An estimated 10 lab classes are conducted in a semester for each lab course. These lab classes are assessed continuously. Each lab experiment is evaluated based on 5 assessment criteria as shown in following table. Assessed performance in each experiment is used to compute CO attainment aswell as internal marks in the lab course.

Grading Criteria	Exemplary (4)	Competent (3)	Needs Improvement (2)	Poor (1)
AC1: Pre-Lab written work (this may be assessed through viva)	Complete procedure with underlined concept is properly written	Underlined concept is written but procedureis incomplete	Not able to write concept and procedure	Underlined concept is not clearly understood
AC2: Writing Modeling	Assigned problem is properly analyzed, correct solution designed, appropriate language constructs/ tools are applied, Program/solution written is readable	Assigned problem is properly analyzed, correct solution designed, appropriate language constructs/ tools are applied	Assigned problem is properly analyzed & correct solution designed	Assigned problem is properly analyzed
AC3: Identification & Removal of errors	Able to identify errors/ bugs and remove them	Able to identify errors/ bugs and remove them with little bit of guidance	Is dependent totally on someone for identification of errors/ bugs and their removal	Unable to understand the reason for errors/ bugs even after they are explicitly pointed out
AC4:Execution & Demonstration	All variants of input /output are tested, Solution is well demonstrated and implemented concept is clearly explained	All variants of input /output are not tested, However, solution is well demonstrated and implemented concept is clearly explained	Only few variants of input /output are tested, Solution is well demonstrated but implemented concept is not clearly explained	Solution is not well demonstrated and implemented concept is not clearly explained
AC5:Lab Record Assessment	All assigned problems are well recorded with objective, design constructs and solution along with Performance analysis using all variants of input and output	More than 70 % of the assigned problems are well recorded with objective, design contracts and solution along with Performance analysis is done with all variants of input and output	Less than 70 % of the assigned problems are well recorded with objective, design contracts and solution along with Performance analysis is done with all variants of input and output	Less than 40 % of the assigned problems are well recorded with objective, design contracts and solution along with Performance analysis is done with all variants of input and output

LAB EXPERIMENTS

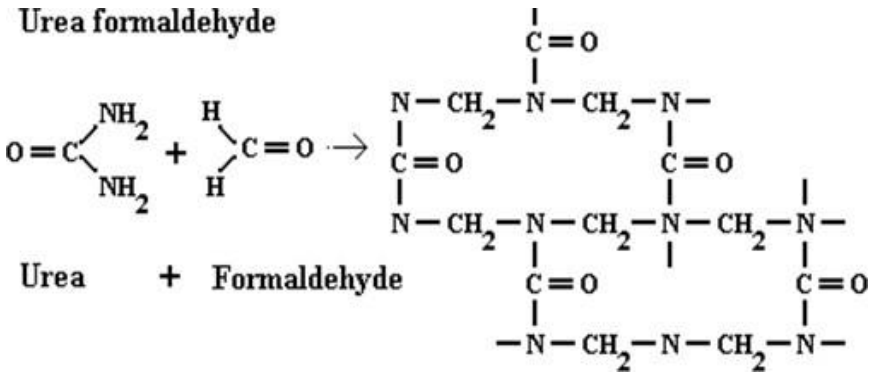
LAB EXPERIMENT 1

AIM: To prepare th**e** Urea-formaldehyde Resin.

REQUIREMENTS:

Urea, formaldehyde solution, conc.H_2SO_4, Distilled water, glass rod, glass funnel, beaker, filter paper, etc.

THEORY: Urea Formaldehyde resin is prepared by condensation reaction between Urea and Formaldehyde in acidic or alkaline medium.



PROCEDURE

1.Take 5 mL of 40% aqueous formaldehyde solution in a 100 mL beaker.

2.To this add 2 g urea powder. Stir with a glass rod to make a saturated solution.

3.Add a few drops of conc. H_2SO_4 and stir vigorously till a white solid mass is formed.

4.Filter the residue and wash it several times with distilled water to remove any acid.

5.Dry the residue in folds of filter paper or in an oven and weigh.

6.Report the yield of urea formaldehyde polymer formed.

OBSERVATION

Weight of empty watch glass = W_1 g

Weight of watch glass + polymer formed = W_2 g

Weight of polymer formed = $W_2 - W_1$ g

RESULT

Weight of urea formaldehyde resin = W g

PRECAUTIONS

1. Keep the face away from the beaker.
2. Add sulfuric acid drop by drop with constant stirring.
3. Wash the solid mass formed several times with water, to remove excess of acid.

VIVA- VOCE QUESTIONS

Q1. What are polymers?
Ans: Polymers are the high molecular weight compounds, made of repeating units called monomers.

Q2. What is the difference between thermoplastic and thermosetting polymers?
Ans: Thermoplastic polymers can be reused whereas thermosetting polymers cannot be reused.

Q3. What are the uses of urea formaldehyde resin?
Ans: Urea formaldehyde resin is used for making plastic parts of electrical appliances, switch boards, as sizing material in paper industry and for making bottle caps and buttons.

Q4. Urea formaldehyde resin is a thermoplastic or thermosetting polymer?
Ans: Urea formaldehyde resin is a thermosetting polymer as it cannot be remolded and reused.

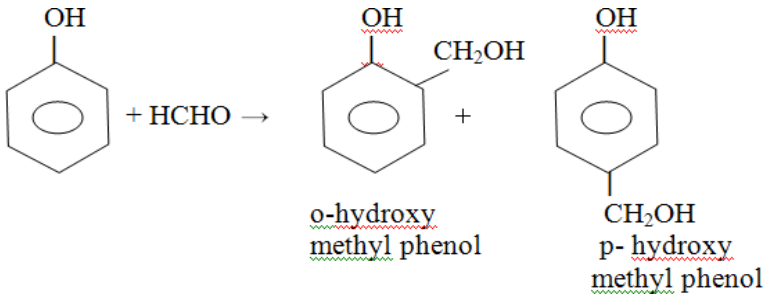
LAB EXPERIMENT 2

AIM: To prepare Phenol formaldehyde (P-F) resin.

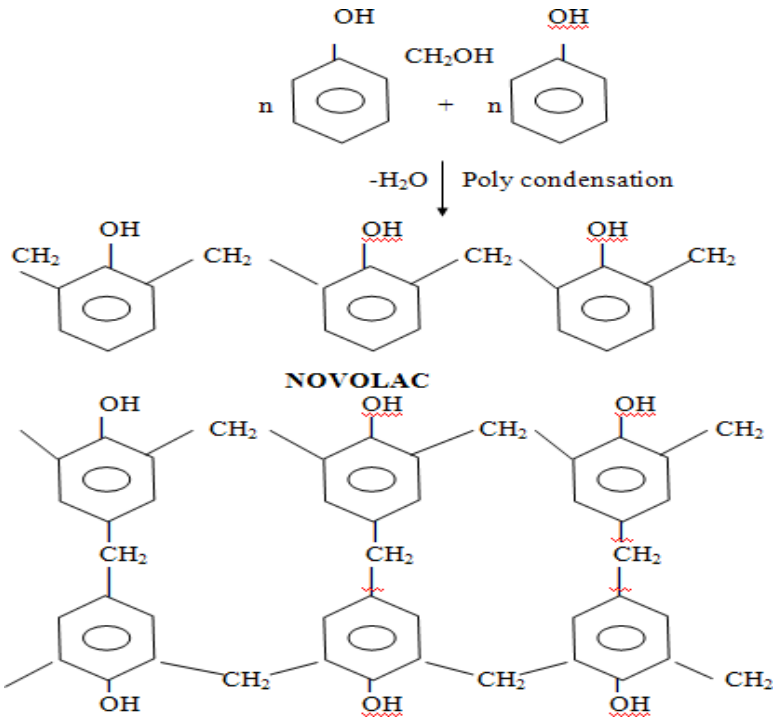
REAGENTS/ APPARATUS: Phenol, 40% formaldehyde solution, conc. HCl, glacial acetic acid, glassrod, glass funnel, beaker, filter paper etc.

THEORY: Bakelite is a copolymer formed by the polymerization of phenol and formaldehyde. It isthermosetting in nature and is also known as PF-resin.

PF- resin is prepared by the condensation of phenol (P) and formaldehyde (F) in presence ofacid catalyst. Phenol first react with ortho and para hydroxy methyl phenol.



Ortho- hydroxy methyl phenol undergoes poly condensation with phenol to form linear chain polymer known as **novolac** which is converted into hard infusible, cross- linked, solid bakeliteon reaction with formaldehyde.



PROCEDURE

1. Place 5 mL of glacial acetic acid and 2.5 mL of 40 % aq formaldehyde solution in a 100mL beaker. Add 2 g phenol safely.
2. Wrap the beaker with a wet cloth or place it in a 250 mL beaker having small amountof water in it.
3. Add conc. HCl drop wise with vigorous stirring by a glass rod till a pink colouredgummy mass appears.
4. Wash the pink residue several times with to make it free from acid.
5. Filter the product and weigh it after drying in folds of a filter or in an oven. Report theyield of polymer formed.

OBSERVATION

Weight of empty watch glass = W_1 g

Weight of watch glass + polymer formed = W_2 gWeight

of polymer formed = $W_2 - W_1$ g

RESULT

Weight of phenol formaldehyde resin = W g

PRECAUTIONS

1. Keep the face away from the beaker.
2. Add sulfuric acid drop by drop with constant stirring as the reaction is exothermic.
3. Do not touch phenol with bare hand as it can burn the skin.
4. Wash the solid mass formed several times with water, to remove excess of acid.

VIVA- VOCE QUESTIONS

Q1. What is degree of polymerization?
Ans: Degree of polymerization refers to the total number of monomeric unit present in a polymer.

Q2. What is the difference between addition and condensation polymer?
Ans: In addition polymers the monomeric units simply add up to form the polymer whereas Condensation polymers are formed by the removal of small molecules like H_2O, H_2 etc.

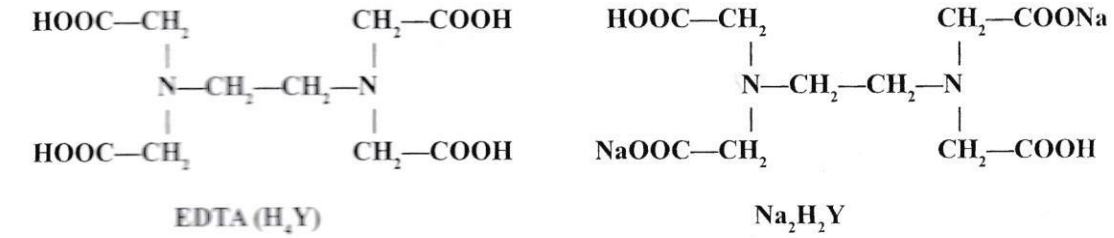
Q3. What is the common name of phenol formaldehyde resin? What are its applications?
Ans: The common name of phenol formaldehyde resin is Bakelite. It is used for making plastic parts of TV, microwave, insulation layer of electric wires, for making chewing gums , used in lacquers.

Q4. Phenol formaldehyde resin is a addition polymer or condensation polymer?
Ans: Phenol formaldehyde resin is a condensation polymer as the polymer is formed by the removal of water molecules.

LAB EXPERIMENT 3

AIM: To determine the hardness of the given water sample by EDTA method.

PRINCIPLE: Hardness of water is due to the presence of calcium and magnesium salts in water. Ethylenediamine tetra acetic acid (EDTA) forms complexes with a large number of cations including Ca^{2+} and Mg^{2+} ions. Hence, total hardness of water can be determined using EDTA.



Since EDTA (free acid) is sparingly soluble its disodium salt Na_2H_2Y, is used for analyticalwork. The disodium salt of EDTA (Na_2H_2Y) has two easily replaceable hydrogen atoms and the resulting ionization may be represented as H_2Y^{2-}.The latter forms complexes with metal ions as follows:

$M^{2+} + H_2Y^{2-} \rightarrow MY^{2-} + 2H^+$ ·····(1)

Where M is Ca^{2+} and Mg^{2+} are present in water. Reaction (1) can be carried out quantitatively at a pH of 10 using Eriochrome black T indicator. Since the reaction involves the liberation of H^+ ions, a buffer mixture is to be used to maintain a pH of 10. The buffer mixture used in the titration is NH_3– NH_4Cl. The hardness of water is usually expressed in terms of ppm (parts per million) of $CaCO_3$.

PROCEDURE:

Determination of total hardness of water sample

Pipette out 25 cm^3 of the given water sample into a clean conical flask. Add 3 cm^3 of NH_3–NH_4C1 buffer and a pinch of Eriochrome black-T indicator. Titrate against EDTA solution tillthe colour of the solution changes from wine red to clear blue. Repeat the experiment for agreeable values.

OBSERVATIONS AND CALCULATIONS

Part A: Preparation of standard solution of disodium salt of EDTA

1. Weight of bottle + EDTA salt = ...g
2. Weight of empty bottle = ...g
3. Weight of EDTA salt transferred = ...g

M

olarity of EDTA = $\frac{\text{Weight of EDTA salt} \times 4}{\text{Molecular weight of EDTA salt}} = \frac{}{\frac{372.2}{4}} \times 4 = \ldots\ldots\ldots\ldots\ldots M$

Part B: Determination of total hardness of water sample

Burette : Standard EDTA solution

Conical flask : 25 cm^3 Hard water sample + 2 cm^3 of NH_3– NH_4C1 buffer

Indicator : Eriochrome black - T

End point : Wine red to clear blue

Burette readings	**Trial I**	**Trial II**	**Trial III**
Final reading			
Initial reading			
Volume of EDTA run down in cm^3			

Concordant burette reading =..... cm^3

$(MV)_{Hard\ Water} = (MV)_{EDTA}$

$M_{Hard\ Water} = \frac{(MV)_{EDTA}}{(V)_{Hard\ Water}} = \frac{\times}{25} = \ldots\ldots\ldots\ldots\ldots M$

Wt/litre of $CaCO_3$ equivalent hardness = $M_{Hard\ Water}$ x Mol. Wt. of $CaCO_3$ (100) = a

$\therefore 10^6 cm^3$ (1 million cm^3) of hard water sample contains $= \frac{a \times 10^6}{1000}$ ppm of $CaCO_3$

Thus total hardness of the given water sample = ppm of $CaCO_3$

RESULT: Total hardness of given water sample is determined as. ..... ppm of $CaCO_3$.

VIVA- VOCE QUESTIONS

Q1. What is hardness of water? Which salts are responsible to cause temporary hardness?
Ans. Hardness is the property of water due to which it does not produce lather with soap. Bicarbonates of calcium and magnesium are responsible to cause temporary hardness.
Q**2.** What do you understand by EDTA?
Ans. Ethylene diamine tetra acetic acid is a chelating agent, which forms stable complex with bivalent Cations like calcium and magnesium.
Q3. Why buffer is required in this titration? Which buffer solution is used in this complexometric titration?
Ans. In this complexometric titration complex between EBT and Ca, Mg and between EDTA and Ca, Mg takes place in alkaline medium, at 9-10 pH. Ammonia buffer (NH_4Cl+NH_4OH) is used to maintain pH in this range.

LAB EXPERIMENT 4

AIM: Determination of viscosity of lubricant by Redwood viscometer (No. 1 / 2).

APPARATUS REQUIRED: Redwood viscometer (No. 1 & 2), stop watch, thermometer, givenlubricant oil and distilled water.

THEORY

Viscosity is defined as the internal friction offered by internal friction offered by the layers of fluid of its flow. Viscosity is a measure of flow ability of a liquid at a definite temperature. It determines the performance of oil under operating conditions. Higher the viscosity of fluid lesser will be its flow.

Coefficient of viscosity is called absolute viscosity is defined as tangential force per unit area required to maintain a unit velocity gradient between two parallel layers a unit apart. It is denoted by η (eta).

$$\text{Mathematically, } \eta = \frac{F / A}{dv/dx}$$

where F = force

A = Area

dv/dx = Velocity gradient

Units: In C.G.S. system : poise = dyne cm^{-2} s

In SI system : N m^{-2} s

The absolute viscosity of lubricant is determined by measuring the time of flow of the oil through a capillary of definite dimensions at uniform temperature. The viscosity is can be measured by a Redwood viscometer.

Description of Redwood Viscometer:

It is available in two sizes. These are:

(i) RW_1 or Redwood No. 1 (Universal)
(ii) RW_2 or Redwood No. 2 (Admirality)

Viscometer	Diameter of capillary	Length of jet
RW_1	1.62 mm	10 mm
RW_2	3.80 mm	50 mm

The rate of discharge of oil through RW_2 is nearly 10 times faster than the dischargethrough RW_1 because receiving flask of RW_2 has a wider mouth.

Construction

It consists of a standard brass oil cup fitted with an agate jet of specific dimension in the middle of base and open at the upper end (Fig. 1). The jet can be opened or closed by a polished ball valve which controls the flow of oil. A pointer is provided in the oil cup to indicate the level up to which oil shall be filled in it. The lid of the cup is provided with a thermometer to note the temperature of oil. Surrounding the oil cup is cylindrical vessel made of copper. This vessel is filled with water and serves as a water bath to maintain the oil at a desired temperature of oil. Itis heated by means of heating coils.

The water bath is provided with stirrer having four blades to maintain uniform temperature of bath. A thermometer is fitted in a water bath to know the temperature of water at its base. A 50 mL flask called Kohlrausch flask is provided below the jet to collect the liquid flowing out of the jet. The apparatus is also provided with leveling screws for its leveling.

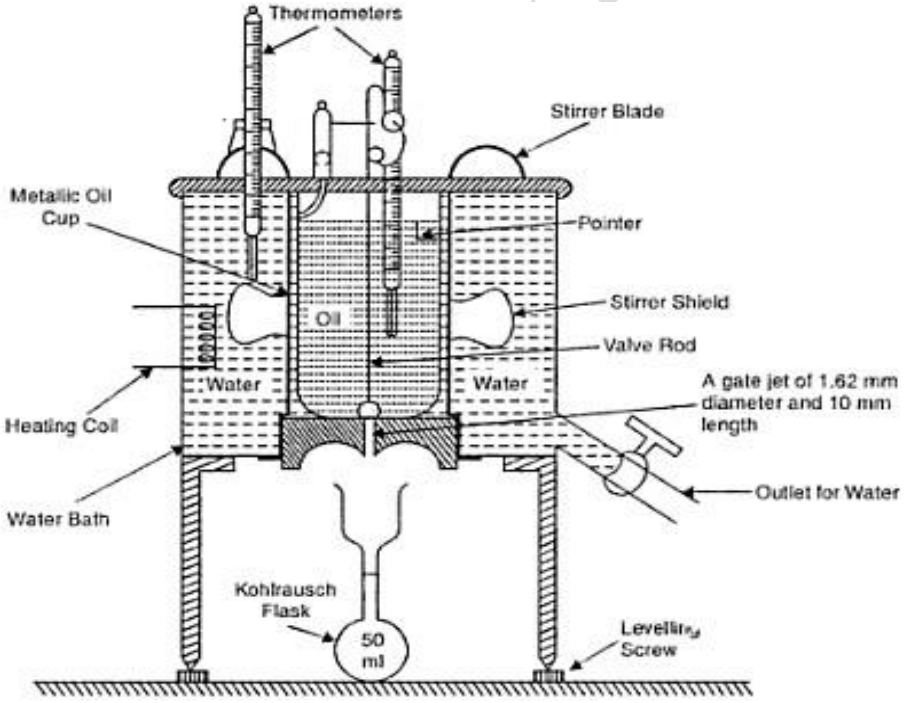


Fig. 1. Redwood apparatus no. 1 & 2.

PROCEDURE

1. Level the viscometer with the help of leveling screws. Fill the outer bath with water and connect to the electric mains. Clean the oil cup and discharge jet with a suitable jet with asuitable solvent like hexane, carbon tetrachloride etc. and properly dry it.
2. Place the ball valve on agate jet to close it. Pour the test oil in the cup carefully up to the pointer. Insert a thermometer and stirrer and cover the lid.
3. Adjust the temp. of water bath until the oil attains the desired temperature. In this period keep water in water bath and oil in oil cup.
4. Place a clean and dry Kohlrausch flask immediately below and directly in the line with the discharge jet
5. Remove the ball when oil attains desired temperature with one hand and start stop watch with the other hand. Allow the oil to flow till the flask is filled up to 50 mL mark. Stopthe stop watch and note the time of flow in seconds.
6. Repeat the experiment 3-4 times and record the reading.
7. Report the mean value in Redwood seconds and also mentioning the viscometer used andthe test temperature.

OBSERVATIONS

Table: For time of flow

Sr. No.	Temperature (^oC)	Time of flow (RW seconds)
1 **2** **3**		

RESULT: Viscosity of given lubricant is …….RW seconds.

PRECAUTIONS

1. The inner cup of the Red wood viscometer must be cleaned with organic solvents and dried before use.

2. Viscometer is required to be leveled properly before use.

3. Stop watch must be on and ball valve must be removed simultaneously.

VIVA- VOCE QUESTIONS

Q1. What is viscosity? What is it's unit in SI ?

Ans. Viscosity is a measure of a fluid's resistance to flow. It's unit in SI system is Pascal second.

Q2. What is the relation between temperature and viscosity?

Ans. Viscosity of a liquid is inversely proportional to temperature, i.e., if the temperature increases viscosity of the liquid decreases.

Q3. What is the difference between Red Wood Viscometer 1 and 2?

Ans. Both the viscometers are similar in structure the only difference is in diameter and length of agate jet.

LAB EXPERIMENT 5

AIM : To prepare a pure sample of iodoform.

Chemicals :
Acetone – 5 ml
Iodine – 5 g
NaOH – 5 %
Methylated spirit

THEORY :

$$CH_3COCH_3 + 4I_2 + 4NaOH \rightarrow CHI_3 + 3NaI + CH_3COONa + 2H_2O$$

PROCEDURE:

- Dissolve 5 g of iodine in 5 ml acetone in a conical flask.
- Add 5 % sodium hydroxide solution slowly with shaking until the colour of iodine is discharged.
- Allow contents of flask to stand for 10 – 15 minutes.
- Filter the yellow precipitate of iodoform through Buchner funnel
- Wash the precipitate with cold water.
- Dry precipitate between the folds of filter paper and weigh it.

RESULT:

Yield of crystals – 5 g
Colour of crystals – yellow
Melting point – 119 ◦C.

PRECAUTIONS:

- Use freshly prepared sodium hydroxide.
- Add sodium hydroxide slowly and with constant stirring.

VIVA- VOCE QUESTIONS

Q1. What does iodoform test indicate?

Ans. The iodoform test reveals the presence of an aldehyde or ketone in which a methyl group is one of the groups immediately connected to the carbonyl carbon. Such a ketone is referred to as methyl ketone. The unknown is allowed to react with a mixture of excess iodine and excess hydroxide in the iodoform assay.

Q2. What is iodoform test used for?

Ans. The methyl ketone reaction of iodine and base is so reliable that the iodoform test (the appearance of a yellow precipitate) is used to test for methyl ketone presence. This is also the case while searching for alpha-position sensitive secondary alcohols containing at least one methyl group.

Q3. How do you do iodoform test?

Ans. Dissolve 5 g of iodine in 5 ml acetone in a conical flask. Add 5 % sodium hydroxide solution slowly with shaking until the colour of iodine is discharged. Allow contents of flask to stand for 10 – 15 minutes. Filter the yellow precipitate of iodoform through Buchner funnel.

LAB EXPERIMENT 6

AIM : To separate the colored component present in the extract of spinach leaves by paper chromatography and find their R_f values

REQUIREMENTS:

Chromatographic chamber with lid
Measuring cylinder
Whatman filter paper strip (10cm.x 20cm)
Glass rod
Fine capillary tube
Isopropyl alcohol
Extract of spinach leaves

THEORY:

In the chromatographic technique, the mixture of substance is applied on a phase called the stationary phase. The stationary phase may be solid or liquid. A moving phase that can be a pure solvent or a mixture of solvents, or a gas is allowed to move slowly over the stationary phase. This moving phase is called the mobile phase. When the mobile phase moves over the mixture on the stationary phase, the components of the mixture gradually separate from one another.

Depending on the basic principle involved in the chromatography, it is mainly classified into two types:

(a) Adsorption Chromatography: It is based on the differential adsorption of the component on the adsorbent (stationary phase). This means that different compounds are adsorbed on an adsorbent at different degrees. The two main types of chromatographic techniques based on the principle of differential adsorption are column chromatography and thin layer chromatography.

(b) Partition Chromatography: The basic principle of partition chromatography is the continuous differential partitioning of components of a mixture between the stationary phase and the mobile phase. The type of Chromatography based on partition chromatography is paper chromatography.

Paper Chromatography:

In paper chromatography, the stationary phase is a special quality paper called chromatography paper. Mobile phase is a solvent or a mixture of solvents. A solution of the mixture is spotted on a line about 2 cm. above the bottom of the paper, called original or base line. Paper is then suspended in a chromatography chamber containing suitable solvent rises up the paper by capillary action and flows over the spot. The paper the paper strips so developed is called ‘chromatogram’. The spots of the separated colorless components may be observed either under ultraviolet light or by the use of an appropriate spray reagent. The distance is travelled by the solvent from original. Line is called solvent front.

The relative absorption of each component of the mixture is expressed in terms of its 'retention factor' (R_f)

$$R_f = \frac{\text{Distance travelled by the component from the original point}}{\text{Distance travelled by the solvent from the original line}}$$

PROCEDURE:

1) Take a whatman filter paper strip and draw a horizontal line 4cm from the lower end of the paper. Then draw another line vertically from the center of the paper. Name the point at which the two lines intersect at P.
2) Put a drop of the extract of spinach leaves at the point P using a fine capillary tube. Let it dry in air.
3) Put another drop on the same spot and dry again (so that the spot is rich in the leaf extract)
4) Pour equal amounts of isopropyl alcohol and distilled water into a chromatographic chamber and mix it well using a glass rod. This is used as the solvent system.
5) Suspend the filter paper vertically in the chromatographic chamber containing the solvent system in such a way that the pencil line remains about 2cm above the solvent level.
6) Close the jar with a lid and keep it undisturbed.
7) Notice the rising solvent system along with the colored components of the leaf extract.
8) After the solvent has risen to about 15cm, we notice two different spots of colored components on the filter paper.
9) Take the filter paper of the jar and using a pencil mark the distance that the solvent system has travelled on the paper. This is called solvent front.
10) Dry the filter paper and put pencil marks at the center of each spot.
11) Measure the distance of each spot from the original line and the distance of the solvent from the original line.

OBSERVATIONS:

Observations can be recorded as:

S.No	Component	Distance travelled by the component from the original line (cm)
1	Orange (Carotene)	
2	Yellow (Xanthophyll)	
3	Light Green (Chlorophyll a)	
4	Dark Green (Chlorophyll b)	

CALCULATION:

Calculate the R_f values of different components of leaf extract by using the formula:

$$R_f = \frac{\text{Distance travelled by the component from the original line}}{\text{Distance travelled by the solvent from the original line}}$$

RESULT:

The R_f values of different components in the extract of spinach leaves are as follows:

1) R_f value of the orange component (carotene) = …….
2) R_f value of the yellow component (Xanthophyll) = …….
3) R_f value of the light green component (Chlorophyll a) = …….
4) R_f value of the dark green component (Chlorophyll b) = …….

PRECAUTIONS

1) Use good quality pencil for drawing the reference line so that the mark does not dissolve in solvent system in which the chromatographic separation is carried out.
2) Always use a fine capillary tube.
3) Keep the jar undisturbed and covered during the experiment.
4) The spot should be small and rich in extract.
5) Allow the spot to dry before putting the strip in the jar.
6) Keep the strip straight. It should not be curled.
7) Do not allow the spot to dip in the solvent.

VIVA- VOCE QUESTIONS

Q1. What is chromatography?
Ans. Chromatography is a process of rapid and efficient separation of components of a mixture**.**

Q2. What are the moving and stationary phases in paper chromatography?
Ans. Water absorbed on cellulose constituting the paper serves as the stationary phase and organic solvent as moving phase.

Q3. What is meant by the term Rf value ?
Ans. Rf (retention factor) of a substance is defined as the ratio of the distance moved up by the solute from

the point of its application to the distance moved up by the solvent from the same point.

Q4. On what factors does the Rf value of a compound depend ?

Ans. The Rf value depends upon following factors

1. Nature of the compound.
2. Nature of the solvent.
3. Temperature.

LAB EXPERIMENT 7

AIM : To determine the surface tension of the given liquid at room temperature bystalagmometer.

REQUIREMENTS: Stalagmometer, Distilled Water, Unknown liquid, Clamp stand, Specific gravity bottle etc.

THEORY:

The surface tension of the given liquid is determined at the room temperature by using stalagmometer. The numbers of drops for the same volume of distilledwater and the given liquid are counted and let these be as n_1 and n_2 respectively. Now if d_1 and d_2 are densities of water and given liquid at the room temperature as determined separately by using specific gravity bottle , then the surface-tension y_2 of the given liquid can be calculated by using the simplified relationship as: $\rho_1/\rho_2 = n_2/n_1 \; d_1/d_2$

PROCEDURE:

1. Clean the stalagmometer and specific gravity bottle thoroughly withchromic acid solution and wash finally with distilled Water and then dry.

2. By immersing lower end in a beaker containing distilled water, suck upwater until it rises above the mark C.

3. Allow the liquid drops to fall and count it.

4. Clean the stalagmometer and dry it. Fill it with liquid it rises above the uppermark C and count the number of drops as before.

5. Clean and dry the specific gravity bottle and measure the density of liquid.

OBSERVATION:

Room Temperature = ^{0}C.

Density of water at room temp. (d_1) = ---------- **gm/cm^3**

Surface tension of water at room temp. (y_1) = **72.14 dynes/cm**

S. No.	Water		Given Liquid	
	No. of drops	Mean (n_1)	No. of drops	Mean (n_2)
1.				
2.				
3.				

CALCULATION:

Measurement of Density of Solution

1. Weight of empty specific gravity bottle = w_1 gm
2. Weight of bottle + water = w_2 gm
3. Weight of bottle + Some volume of

liquid = w_3 gmDensity of the given liquid (d_2)

= ($w_3 - w_1$/ $w_2 - w_1$) X d_1 Also, $\rho_1/\rho_2 = n_2/n_1$

$\times d_2/d_1$.

$\rho_2 = \rho_1 \times n_1/n_2 \times d_2/d_1$.

Where ρ_1 = surface tension of water at room temperature.

Substituting the values of ρ_1, n_1, n_2, d_1 and d_2 the surface- tension of the given liquid at roomtemperature thus becomes known.

RESULT: The surface tension of liquid at room temperature is (dynes/cm).

PRECAUTIONS

1. The stalagmometer and specific gravity bottle should be cleaned properly and dried before use.

2. Fit the stalagmometer vertically.

3. The rate of the fall of drops should be adjusted in a way of having interval of at last 2-3 seconds in successive drops. The number of drops per minute must bein between 15-20.

4. The drops should fall from the tip of the stalagamometer under their own weight rather than pushing them by force.

5. Wash and dry the stalagmometer after use.

VIVA-VOCE QUESTIONS

Q1. What is surface tension?
Ans. Surface tension is the force per unit length working on the surface of a liquid due to the cohesive forces of the molecules of the liquid.

Q2. What is the unit of surface tension?
Ans. The SI unit of surface tension is Newton M^{-1} . In CGS system it is Dynes cm^{-1} .

Q3. What happens to the surface tension of water if soap is mixed in it?
Ans. Surface tension of water decreases as the attraction between the water molecules decreases.

LAB EXPERIMENT 8

AIM : To determine the chloride content in supplied water sample by Mohr's method.

REQUIREMENTS: Burette, Pipette, Conical Flask, Beaker, Funnel $AgNO_3$ (N/100), Watersample, Potassium chromate indicator.

THEORY: Mohr's method is used to determine chloride content in water sample. In this method a slightly alkaline solution is titrated against standard silver nitrate solution using potassium chromate as an indicator. As the titration proceeds, the chloride ions present react with $AgNO_3$ forming white precipitate of AgCl. The extra drop of $AgNO_3$ reacts with indicator forming red silver chromate. The change of colour from bright yellow to faint but distinct reddish brown colour marks the end point.

$NaCl + AgNO_3 \rightarrow AgCl\downarrow + NaNO_3$

White ppt.

$2AgNO_3 + K_2CrO_4 \rightarrow Ag_2CrO_4\downarrow + 2KNO_3$

Reddish brown

Reddish colour disappears if solution contains high concentration of chloride ions.

$Ag_2CrO_4 + 2Cl^- \rightarrow 2AgCl\downarrow + CrO_4^{--}$

In this method a slightly alkaline solution is used because in acidic medium Ag_2CrO_4 gets dissolved where as in basic medium AgOH gets formed.

END POINT:bright yellow colour to reddish brown colour

PROCEDURE:

1. Fill the burette with $AgNO_3$ Solution.
2. Note down the initial reading of the burette.
3. Pipette out 10mL of the water sample in a clean conical flask.
4. Add 2 drops of potassium chromate indicator and titrate against standard $AgNO_3$ with constant shaking until a color change from yellow to reddish brown color marks the end-point.
5. Note the volume of the $AgNO_3$ from the burette reading.
6. Repeat the titration for concordant readings.
7. Repeat the titration for blank correction. Note down initial reading of the burette.
8. Pipette out 10mL of the water sample in a clean conical flask.
9. Add 2 drops of potassium chromate indicator and titrate against standard with constant shaking until a color change from yellow to reddish brown color marks the end point.
10. Note the volume of the $AgNO_3$ from the burette reading.
11. Repeat the titration for concordant readings.
12. Repeat the titration for blank correction.

OBSERVATION:

Table for sample solution

S.No.	Volume of water sample taken / mL	Burette reading / mL		Volume of $AgNO_3$ $\mathbf{V_2}$ (mL)
		Initial	Final	
1	10			
2	10			
3	10			

Calculations: Chloride content determination :

$N_1 V_1 = N_2 V_2$

(Sample) $(AgNO_3)N_1 \times 25 = N_2 \times V_2\ N_1 = N_2 \times V_2/ 10$

So strength of chloride ions = $N_1 \times 35.5$ g/ L

$= N_1 \times 35.5 \times 1000$ mg/ L

Result: The chloride content in given water sample =….. mg/L.

Precautions

1. The apparatus should be washed with distilled water.
2. The reaction mixture should be properly shaken during titration.
3. End point should be noted carefully.

VIVA-VOCE QUESTIONS

Q1. What kind of titration is used in determination of chloride content in the given water sample?
Ans. In determination of chloride content in the given sample of water precipitation titration method is used.

Q2. Which indicator is used in this method of chloride determination?
Ans. In this method of chloride determination potassium chromate (K_2CrO_4) is used as indicator.

Q3. What color you obtain at the end point? How the color is formed?
Ans. The solution turns brown at the end point in this method of chloride determination. The brown color is formed due to the formation of brown colored precipitates of silver chromate(Ag_2CrO_4)

LAB EXPERIMENT 9

AIM: Determination of strength of HCl solution by titrating it against NaOH solution conductometrically.

APPARATUS REQUIRED: conductivity meter, conductivity cell

Chemicals required: $\frac{N}{100}$ KCl, $\frac{N}{10}$ NaOH, $\frac{N}{100}$ HCl and distilled water.

Theory:

End point of a volumetric analysis can also be found by conductometric titration which involves measurement of conductance of solution during titration. The principle of these titrations is that electrolytic conductance varies during the course of titration as it depends upon ions in solution and their mobility. The end point is found to from a plot of conductance ofvalues against volume of titrant added which gives two lines intersecting each other. The point ofintersection gives the end point of titration.

Titration of HCl vs NaOH (strong acid vs strong base) is studied by titrating it a known volume of acid against standard alkali and measuring conductance of solution at different times. Initially the conductance is high as HCl is strong electrolyte and is highly ionized. On adding NaOH from burette, the conductance decreases, it is because fast moving H^+ ions of HCl are neutralized and replaced by slow moving Na^+ ions.

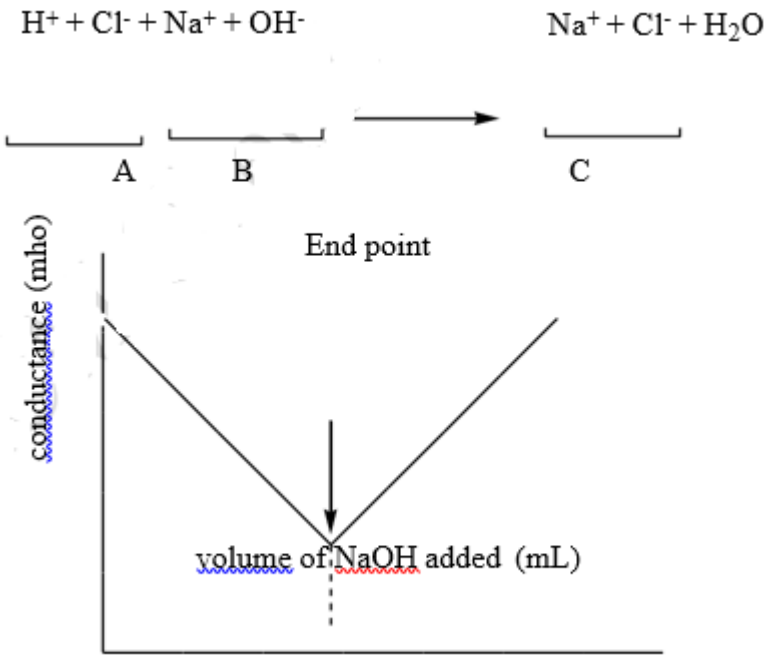


Fig. 1 plot of conductance vs volume of NaOH added

After complete neutralization, added NaOH increases the conductance of solution due to addition of highly mobile OH^- ions, conductivity is minimum at equivalent point. On plotting conductance vs vol. of NaOH added, a V-shaped graph is obtained (Fig 1). The point ofintersation of two lines gives end point. From volume of NaOH used at end point, the strength ofHCl solution can be determined.

Procedure

1. Calibrate the conductivity meter using N/10 KCl.
2. Pipette out 50 mL of given HCl solution in a 100 mL beaker.
3. Dip conductivity cell in a HCl solution.
4. Note down the conductance value of solution.
5. Add 1 mL of N/10 NaOH solution from burette. Stir solution with a glass rod and notedown the conductance of solution when it becomes stable.
6. Add NaOH solution in 1 mL lots and note the corresponding conductance values till conductance becomes constant.
7. Plot a graph between conductance (Y-axis) and volume of added NaOH solution (alongX-axis).
8. Find out the volume of NaOH used at end point of intersation of two lines in graph.

Observation

Temperature of HCl solution = …..^oC

Volume of HCl taken = 50 ml Normality of

NaOH taken = N/10

Table: Titration of HCl vs NaOH solution

S. No.	Vol. of NaOH added (mL)	Observed conductance (mho)
1 2 3 . .	0 1 2 . .	

General calculations

Let the volume of NaOH used at end point (from graph) = V mL

Applying normality equation

$$N_1V_1 = N_2V_2 \text{ (NaOH)}$$

$$N_1 \times 50 = (1/10) \times V$$

$$N_1 = (1/10) \times (1/50) \times V$$

Strength of HCl solution = normality × equivalent wt of HCl

$$= \qquad = X \text{ mg } L^{-1}$$

Result: Strength of HCl solution is ….. mg L^{-1}

Viva-voce Questions

Q1. What is the basic principle of conductometric titration?

Ans. The conductivity of the electrolytic solution changes with the change in concentration of ions, by continuous careful monitoring this conductivity change we can find out the end point of the reaction.

Q2. What are factors affecting conductometric titration?

Ans. The following factors affect the conductometric titration:

(vi) Number of ions

(ii) Size of ions

Q3. In the titration curve the conductivity of the solution first decreases then increases. Why?

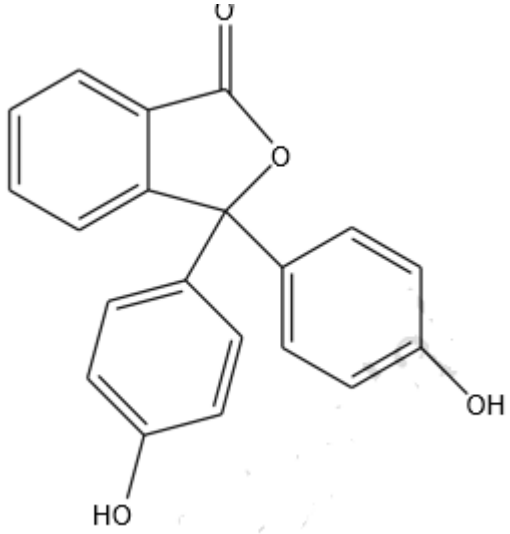
Ans. In the titration curve initially conductivity decreases due to decrese in H^+ . After equivalence point it increases due to increase in number of OH^- .

LAB EXPERIMENT 10

Aim: To find out saponification number of oil.

Chemical required: Standard alc. $\frac{N}{2}$KOH solution, standard alc. $\frac{N}{2}$HCl solution, ethyl methylketone as solvent.

Indicator: Phenolphthalein



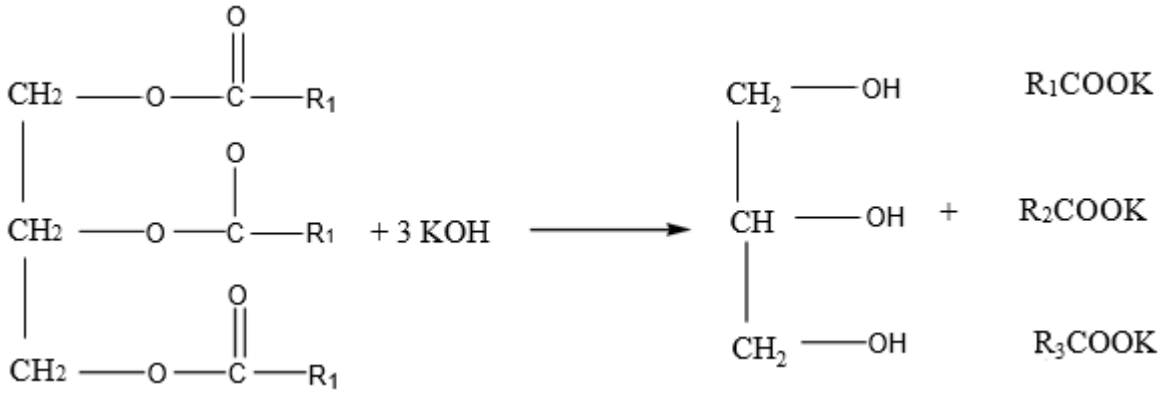
Phenolphthalein**End**

point: Disappearance of pink colour **Theory**

Saponification is process of alkaline hydrolysis of oils (vegetable or animal) and fats giving soap.

Saponification number is defined as number of milligrams of KOH required to sponify 1 mg of afatty oil.

For determination of sponification number of an oil, a known weight of oil is refluxed with a known excess of standard alc. KOH in a suitable solvent. During refluxing sponificationof oil takes place.



(R_1, R_2, R_3 may be same or different)

Amount of unreacted KOH left behind is determined by titration of mixture against standard HCl using phenolphthalein indicator. Disappearance of pink colour of solution marks the end point.

$H^+ + OH^- \rightarrow H_2O$

A blank experiment without oil is also performed. From the volume of HCl used in two titrations, saponification of oil can be obtained.

Procedure

1. Take two 250 mL conical flasks and lebel them as I and II. Weigh accurately about 1-2 gm of given oil in a weighing bottle and transfer it to flask I. Weigh the empty bottle also.
2. Add 25 mL of ethyl methyl ketone and 25 mL of alc. KOH solution to both flaskas.
3. Put an air condenser in mouth of each bottle of each flask and keep them for refluxing on a water bath for 45 minutes.
4. Remove the flask from water bath. Wash the inner walls of each condenser with some distilled water into the respective flasks. Remove both condenser and cool both the flasks at room temperature.
5. Add 7-8 drops of phenolphthalein indicator to each flask. Titrate the solution of eachflask against N/2 HCl taken in a burette till pink colour just disappears. Note burette reading in both titrations.

Observations

Weight of weighing bottle + oil sample = W_1 g
Weight of empty weighing bottle = W_2 g Weight of
oil taken for experiment = $W_1 – W_2$ g
Volume of ethyl methyl ketone added to each flask = 25 mL
Volume of N/2 alc. KOH added to each flask = 25 mL

Table: Titration of unreacted KOH vs N/2 HCl

	Initial burette reading (mL)	Final burette reading (mL)	Volume of HCl used (mL)
For flask I			
For flask II			

Volume of N/2 HCl used by unreacted KOH in flask I = A mL

Volume of N/2 HCl used by unreacted KOH in flask II = B mL

Vol. of N/2 HCl is equivalent to volume of N/2 KOH used for sponification of $W_1 - W_2$ goil = (B-A) mL

$$\text{Saponification number of oil} = \frac{\text{Vol. of KOH used} \times \text{normality of KOH} \times \text{Eq. Wt. of KOH}}{\text{Wt. of oil sample}}$$

$$= \frac{(B-A) \times 1/2 \times 56}{W_1 - W_2} = \frac{(B-A) \times 28}{W_1 - W_2} = X$$

Result: Saponification number of given oil is X.

Viva-voce Questions

Q1. What is the formula for saponification value?

Ans. The formula for saponification value is SV = (Molecular weight of fat / 56.1) x 1000.

Q2. What is the significance of saponification value?

Ans. Saponification value is used to determine the average molecular weight of the fatty acids present in a sample of fat. It is also used to determine the purity of a fat sample.

Q3. What is the difference between acid value and saponification value?

Ans. Acid value measures the amount of free fatty acids present in a sample of fat, while saponification value measures the average molecular weight of the fatty acids present in a sample of fat.

Chemistry Lab (BSC-101P)

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Crosschecked By
HOD Applied
Science

Please spare some time to provide your valuable feedback.