DRONACHARYA
Group of Institutions

LABORATORY MANUAL
ENGINEERING CHEMISTRY LAB
SUBJECT CODE: BAS - 152/252
B. TECH. (First Year Applied Science) SEMESTER-I/II

Academic Session:

Student Name:	
Roll. No.:	
Branch/Section:	

Dronacharya Group of Institutions
Plot No.27, Knowledge Park-3,Greater Noida,Uttar Pradesh 201308

Affiliated to

Dr.APJ Abdul KalamTechnical University
Lucknow, Uttar Pradesh 226031

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Vision of the Institute

"Instilling core human values and facilitating competence to address global challenges by providing Quality Technical Education."

Mission of the Institute

M1 - Enhancing technical expertise through innovative research and education, fostering creativity and excellence in problem-solving.

M2 - Cultivating a culture of ethical innovation and user-focused design, ensuring technological progress enhances the well-being of society.

M3 - Equipping individuals with the technical skills and ethical values to lead and innovate responsibly in an ever-evolving digital landscape.

Vision of Applied Science Department

- To inculcate a strong foundation in budding technocrats in the field of basic sciences and technology empowering them to learn engineering better and contribute to make a better world.

Mission 0f Applied Science Department

M1: To provide a strong foundation of knowledge and practical skills enabling technocrats to utilize scientific principles to give solutions to complex engineering problems.

M2: To guide students towards self-directed learning, self-discipline, and active engagement through innovative teaching and learning approaches.

M3: To inculcate values and ethics in students and make them responsible citizens of India.

Programme Educational Objectives (PEOs)

PEO1: Students basic concepts in applied science will be enhanced that is necessary for success in industry or in engineering practices as well as advanced study.

PEO2: Students will be equipped with problem-solving, laboratory, and design skills essential for technical careers focused on addressing critical challenges.

PEO3: Students will possess the ability to maintain the environmental serenity while adapting to the dynamic changes in the industry.

Programme 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 ofmathematics, 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 researchmethods including design of experiments, analysis and interpretation of data, and synthesisof 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 leaderin 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 andreceive 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 engagein independent and life-long learning in the broadest context of technological change.

University Syllabus

Part- A: Instrumental Experiments

1. Determination of pKa value of a weak acid using pH meter.
2. Estimation of FAS present in the given solution by potentiometric method
3. Determination of amount of HCl and CH3COOH present in a mixture by conductometry
4. Estimation of copper in the effluent from electroplating industry by colorimetric method.

Part-B: Volumetric Experiments

1. Determination of Chemical oxygen demand of industrial waste water
2. Determination of percentage of copper in brass by Iodometric method
3. Determination of percentage of iron in the given rust solution using standard Potassium Dichromate solution (External indicator method)
4. Determination of Total hardness of given water sample by rapid EDTA method
5. Determination of Nickel using EDTA by complexometric method

Part-C: Virtual & Demonstration Experiments

1. Determination of Viscosity coefficient of a liquid using viscometer (Virtual Experiment)
2. Determination of calorific value of solid fuel using bomb calorimeter (Demonstration)
3. Synthesis of ZnO nanomaterial by sol-gel method (Demonstration)

Course Outcomes

CO 1	Estimate the amount of substance present in the given solution using colorimeter, potentiometer and conductivity meter.
CO 2	Examine the total hardness and chemical oxygen demand in the given solution by volumetric analysis method
CO 3	Evaluate the percentage of copper, Nickel and Iron in the given analyte solution.
CO 4	Determine the calorific value of fuel, pKa and coefficient of Viscosity of a given sample.

Lab Rule of conduct DO's and DON'Ts

1. Conduct yourself in a responsible manner at all times in the laboratory. Don't talk aloud or crack jokes in lab.
2. A lab coat should be worn during laboratory experiments. Dress properly during a laboratory activity. Long hair, dangling jewellery and loose or baggy clothing are a hazard in the laboratory.
3. Observe good housekeeping practices. Replace the materials in proper place after work to keep the lab area tidy.
4. Do not wander around the room, distract other students, startle other students or interfere with the laboratory experiments of others.
5. Do not eat food, drink beverages or chew gum in the laboratory and do not use laboratory glassware as containers for food or beverages. Smoking is strictly prohibited in lab area.
6. Do not open any irrelevant internet sites on lab computer
7. Do not use a flash drive on lab computers.
8. Do not upload, delete or alter any software on the lab PC

Rules & Guidelines for conducting Lab-Work

1. Students are not allowed to touch any equipment, chemicals or other materials in the laboratory area until you are instructed by Teacher or Technician.
2. Before starting Laboratory work follow all written and verbal instructions carefully. If you do not understand a direction or part of a procedure, ASK YOUR CONCERN TEACHER BEFORE PROCEEDING WITH THE ACTIVITY.
3. Before use equipment must be read carefully Labels and instructions. Set up and use the equipment as directed by your teacher.
4. If you do not understand how to use a piece of equipment, ASK THE TEACHER FOR HELP!
5. Perform only those experiments authorized by your teacher. Carefully follow all instructions, both written and oral.
6. Unauthorized experiments are not allowed in the Laboratory.
7. Students are not allowed to work in Laboratory alone or without presence of the teacher.
8. Any failure / break-down of equipment must be reported to the teacher.
9. Protect yourself from getting electric shock.

Lab Safety Guidelines

1. Obtain authorization from the lab In charge prior to entering the lab working area. Smoking is strictly prohibited inside the Lab area.
2. Ensure that safety devices are adequate, appropriate and in good working order.
3. Wear the appropriate personal protective equipment when conducting work with hazardous materials or procedures.
4. Eye protection should be worn when performing tasks with potential to generate flying particles or debris.
5. Most power tool related tasks generate such hazards.
6. Every Student should know the locations and operating procedures of all safety equipment including, First AID KIT (s) and Fire extinguisher.
7. Know where the fire alarm and the exits are located.
8. Laboratories must be locked if no one is in the lab.
9. Proper handling and disposal of bio-hazardous materials, including all patient specimens.

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 firewood 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 the 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.

Precautions (In case of Fire)

1. Turn the equipment off. If the 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 covering it 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.

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.

LIST OF EXPERIMENTS

1. Determination of alkalinity in the given water sample.

2. Determination of temporary and permanent hardness in water sample using EDTA as standard solution.

3. Determination of available chlorine in bleaching powder.

4. Determination of chloride content in water sample by Mohr's method.

5. To determine the iron content in the given sample by redox titration using external indicator.

6. Determination of pH by pH-metric titration.

7. Viscosity of kerosene by Redwood Viscometer No-1.

8. Verification of Beer's law.

9. Determination of surface tension of a given liquid.

10. Preparation of Bakelite and urea formaldehyde resin.

Left Hand Side with Pencil

Date

Aim:

Observation Table:

S. No	Volume in the Titration Flask (ml)	Burette Reading (ml)		Volume of Solution used
		Initial	Final	
1				
2				
3				

(This table is only for volumetric titrations)

Graph if required:

Calculations:

Result:

Right Hand Side with Pen (blue only)

Date:

Object:

Apparatus:

Chemicals:

Theory:

Procedure:

Precautions:

Result:

EXPERIMENT NO:- 1

OBJECT: To determine the alkalinity of given water sample by neutralization titration.

APPARATUS: Beaker, Pipette, Burette, Conical Flask

CHEMICALS: Alkalinity Sample, 0.1N HCl, Methyl Orange and Phenolphthalein.

THEORY: The alkalinity in water is due to the presence of hydroxyl ion (OH^-), carbonate ion (CO_3^{2-}) and bicarbonate ion (HCO_3^-) present in the given sample of water. These can be estimated separately by titration against standard acid, using phenolphthalein and methyl orange indicators. The chemical reaction involved can be shown by the equations given below:

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

ii) $CO_3^{2-} + H^+ \rightarrow HCO_3^-$

iii) $HCO_3^- + H^+ \rightarrow H_2O + CO_2$

PROCEDURE: Take 25ml of the sample solution in conical flask with help of a pipette. Add 2-3 drop of phenolphthalein indicator. Titrate this sample against HCl solution until the pink colour caused by phenolphthalein just disappears. Note down this reading as phenolphthalein end point. Now add 2-3 drops of methyl orange indicator in the same solution. Continue the titration until yellow colour change into pinkish orange. Note the volume of acid used. This is methyl orange end point.

OBSERVATION: Normality of HCl solution = N/10

cator used : nolphthalein

point: k→ Colorless

cator used : hyl Orange

point: low→ Pinkish orange.

OBSERVATION TABLE:

For Phenolphthalein Indicator

o	ume of alkalinity sample he Titration Flask (ml)	ette Reading (ml)		ume of N/10HCl Solution
		al	l	

Volume of N/10HCl Solution used from burette (ml) to neutralize25 ml of alkalinity sample $=V_1$ml

For Methyl Orange Indicator

. No	olume of alkalinity sample the Titration Flask (ml)	urette Reading		olume of N/10HCl Solution sed from burette (ml)
		itial	inal	

Volume of N/10HCl Solution used from burette (ml) to neutralize 25 ml of alkalinity sample = V_2ml
(This is total volume starting with pink colour to colourless and then yellow to pinkish orange)

CALCULATIONS:

Volume of water sample solution taken in titration flask = 25 ml. Volume of HCl used for Phenolphthalein end point = V_1ml Volume of HCl used for methyl orange end point = V_2ml

Equivalents of OH^- + equivalents of ½ CO_3^{2-} = Equivalent of HCl in V_1 of N/10 HCl –(1)
Equivalents of OH^- + equivalents of CO_3^{2-} = Equivalent of HCl in V_2 of N/10 HCl –(2)

Solving equation (1) & (2)

Equivalents of CO_3^{2-} = $2(V_2 - V_1)$ of N/10 HCl
Equivalents of OH^- = $(2V_2 - V_1)$ of N/10 HCl

1) Calculation of alkalinity due to OH^-

Strength of OH^- = $(2V_2 - V_1)/(25\times10)$ equivalent/L
Strength of OH^- in terms of $CaCO_3$ = $(2V_2 - V_1)/250)\times50\times1000$ mg/l

2) Calculation of alkalinity to Na_2CO_3

Strength of CO_3^- = $2(V_2 - V_1)/25\times10$ equivalent/L= $2(V_2 - V_1)/250\times50\times1000$ mg/l

RESULT:

Alkalinity due to NaOH = ppm.

Alkalinity due toNa_2CO_3 ion = pm.

Total Alkalinity = ppm

PRECAUTONS:

1. All the solutions should be freshly prepared
2. In each titration same amount of indicator should be added
3. There should not be any leakage from burette
4. While taking reading from burette, eye should be parallel to it.
5. Before using any solution, shake it properly

QUIZ QUESTIONS

1. What is alkalinity?
2. Why OH^- and HCO_3^- ions cannot exist together?
3. Express all different types of alkalinity
4. Why do we use two indicators to find the alkalinity in given water sample?

EXPERIMENT NO:- 2

OBJECT: To determine the temporary and permanent hardness of a given water sample by complexometric titration using EDTA.

APPARATUS:- Burette, Pipette, Conical Flask, Beaker Measuring cylinder.

CHEMICALS: N/100 EDTA Solution, Water sample, Eriochrome Black-T, Buffer Sol. (pH = 10)

THEORY: When Eriochrome black T (indicator) is added to hard water solution at around pH 10, it forms wine red colored unstable complex with Ca^{2+} and Mg^{2+} ions of the sample water. When this wine red colour solution is titrated against EDTA solution, the colour of the solution changes from wine red to blue colour at the end point.

Ethylene diamine tetraactic acid (EDTA) is a well knowing complexing agent which is widely used in analytical work, on account of its powerful complexing action and commercial availability.

PROCEDURE: Take 25 ml of hard water sample in a conical flask with help of a pipette. Add 5 ml of buffer solution. Add 5 drops of Eriochrome black-T indicator. Colour of the solution turns wine red. Titrate the solution against EDTA until the colour changes from wine red to blue. Repeat the titration for two concordant readings. This reading corresponds to total hardness.
Take about 250 ml of the hard water in 500 ml beaker and boil it for half an hour, cool it and titrate the solution as mentioned above for permanent hardness. Repeat the titration for two concordant readings

OBSERVATION:

Normality of EDTA solution = N/100

1. Indicator used Erichrome Black- T
   End point: Wine red → Blue

OBSERVATION TABLE:

For Tap Water

S.No	Volume of tap water in the Titration Flask (ml)	Burette Reading (ml)		Volume of N/100EDTA Solution used from burette (ml)
		Initial	Final	
1				
2				
3				

Volume of N/100EDTA Solution used from burette (ml) to neutralize 25 ml of water sample = $v_2$ml

For Boiled Water

S.No	Volume of boiled water in the Titration Flask(ml)	Burette Reading(ml)		Volume of N/100 EDTA Solution used from burette(ml)
		Initial	Final	
1				
2				
3				

Volume of N/100EDTA Solution used from burette (ml) to neutralize25 ml of boiled water = $v_3$ml

CALCULATION:

a) Calculation of Total Hardness :-
Equivalents of Ca^{+2}&Mg^{+2} in the water sample = Equivalents of EDTA consumed.

$$N_1V_1=N_2V_2$$
(water) (EDTA)
$$N_1= (1/100xv_2ml)/25ml$$

Strength of total hardness causing ions in terms of $CaCO_3$ ions = N1x50 gm/litre

$$= N_1\times50 \times1000mgm/litre \text{ (in terms of calcium carbonate)}$$
$$= N_1\times50\times1000ppm$$

b) Calculation of Permanent Hardness :-
Equivalents of Ca^{+2}&Mg^{+2} in the water sample = Equivalents of EDTA consumed.

$$N_1V_1=N_2V_2$$
(water) (EDTA)
$$N_1= (1/100\times V_3ml)/25ml$$

Strength of Permanent Hardness hardness causing ions in terms of $CaCO_3$ ions
= N_1x50 gm/litre

$$= N_1\times50 \times1000mgm/litre \text{ (in terms of calcium carbonate)}$$
$$= N_1 \times50\times1000ppm$$

c) Calculation of Temporary Hardness = Total Hardness-Permanent Hardness

RESULT:
Total Hardness S_1 =_____ppm
Permanent Hardness S_2 =_____ppm
Temporary hardness $S_1 - S_2$ =_____ppm

PRECAUTONS:

1. All the solutions should be freshly prepared
2. In each titration same amount of indicator and buffer solution should be added
3. There should not be any leakage from burette
4. While taking reading from burette, eye should be parallel to it.
5. Before using any solution, shake it properly

QUIZ QUESTIONS

1. What do you understand by the term hardness and how many types of hardness is there?
2. Why hardness is expressed in terms of $CaCO_3$ equivalents.
3. What is EDTA. Explain the mechanism of color change from wine red to blue.
4. Explain the difference between temporary and permanent hardness

EXPERIMENT NO:- 3

OBJECT: To determine the percentage available chlorine in given bleaching powder sample.

APPARATUS: Beaker, Pipette, Burette, Conical Flask, Measuring Cylinder

CHEMICALS: Bleaching powder sample, potassium iodide, dilute acetic acid, N/20 sodium thiosulphate solution, freshly prepared starch solution

THEORY: The amount of chlorine liberated by the action of dilute acids on bleaching powder is termed as available chlorine and expressed as percentage weight of bleaching powder. When dilute acetic acid reacts with bleaching powder than free chlorine is liberated. The liberated chlorine reacts with potassium iodide solution to give free iodine. This liberated free iodine is than titrated against n/20 sodium thiosulphate solution using freshly prepared starch solution.

$CaOCl_2 + 2CH_3COOH \rightarrow (CH_3COO)_2Ca + H_2 + Cl_2$

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

(Liberated iodine)

$Starch + I_2 \rightarrow$ Starch iodine complex

(Dark blue colour)

$I_2 + 2NaS_2O_3 \rightarrow Na_2S_4O_6 + 2NaI$

PROCEDURE: Weighed 3 gm bleaching powder is dissolved in 250ml of tap water and shaken properly to obtain a homogeneous solution. Fill the burette after rinsing with hypo solution and note down initial burette reading. Pipette out 10ml of bleaching powder solution in 100ml titration flask. Add 3gm solid KI and 5ml of acetic acid, solution becomes dark brown.Titrate liberated iodine against sodium thiosulphate solution till light yellow colour persists. Add 6-7 drops of freshly prepared starch solution. Solution turns blue. Continue adding hypo solution till blue colour disappears. Repeat the same process to get three concordant readings.

OBSERVATION:

Normality of Sodium thiosulphate solution = N/20

1. Indicator used: Freshly prepared starch solution
   End point: Blue → Colorless

OBSERVATION TABLE:

S. No	Volume of bleaching powder solution in the Titration Flask (ml)	Burette Reading (ml)		Volume of N/20 sodium thiosulphate Solution used from burette(ml)
		Initial	Final	
1				
2				
3				

Volume of N/20 sodium thiosulphate Solution used from burette (ml) to neutralize10 ml of bleaching powder sample solution =$v_2$ml

CALCULATION:

$$N_1V_1 = N_2V_2$$

(Bleaching powder solution) (Hypo solution)

$$N_1 = (1/20 \times V_2\text{ml})/10\text{ml}$$

Strength in terms of available chlorine = $N_1 \times 35.5$ gm/litre
250 ml of sample contains = 3gms of bleaching powder
% available chlorine in the given sample of bleaching powder= $(N_1 \times 35.5 \times 3 \times 100)/250$

RESULT: Percentage available chlorine in given sample of bleaching powder is----------

PRECAUTONS:

1. Starch solutions should be freshly prepared
2. In each titration same amount of indicator, KI and acetic acid should be added
3. There should not be any leakage from burette
4. While taking reading from burette, eye should be parallel to it.
5. Lumps of bleaching powder should be powdered before making solution

QUIZ QUESTIONS

1. Why is a fresh solution of starch used?
2. What is the difference between available chlorine and chlorine content in bleaching powder?
3. Why is starch indicator added near the end point?
4. Why blue colour disappears at the end point in iodine titration?

EXPERIMENT NO:4

OBJECT: To determine the chloride content in given water sample by Mohr's method.

APPARATUS: Burette, Pipette, Conical Flask and Beaker.

CHEMICALS: Standard Silver Nitrate ($AgNO_3$) solution, Water sample, Potassium chromate (K_2CrO_4) indicator

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

$$NaCl + AgNO_3 \rightarrow \underset{\text{White ppt.}}{AgCl} + NaNO_3$$

$$2AgNO_3 + K_2CrO_4 \rightarrow \underset{\text{Reddish brown}}{Ag_2CrO_4} + 2\,KNO_3$$

PROCEDURE: Pipette out 10 ml of water sample in a conical flask. Add 5 drops of freshly prepared K_2CrO_4 solution. Titrate it against standards $AgNO_3$ solution until the brick red colour persists. Repeat the titration till two concordant readings are obtained.

OBSERVATION:

Normality of $AgNO_3$ solution = N/50

1. Indicator used — Freshly prepared K_2CrO_4 solution
   End point: — colourless → Brick red precipitates

OBSERVATION TABLE:

S.No	Volume of water sample in the Titration Flask(ml)	Burette Reading(ml)		Volume of N/50$AgNO_3$Solution used from burette(ml)
		Initial	Final	
1				
2				
3				

Volume of N/50$AgNO_3$Solution used from burette (ml) to neutralize10 ml of water sample =V_2ml

CALCULATION:

$$N_1V_1 = N_2V_2$$
$$(\text{water sample})\ (AgNO_3 \text{ solution})$$

$$N_1 = (1/50 \times v_2\text{ml})/10\text{ml}$$

Strength in terms of chloride content = $N_1 \times 35.5$ gm/litre
$= N_1 \times 35.5 \times 1000$mgm/litre
$= N_1 \times 35.5 \times 1000$ppm

RESULT: Amount of chloride content in given water sample = ---------- ppm

PRECAUTONS

1. In each titration same amount of indicator should be added
2. There should not be any leakage from burette
3. While taking reading from burette, eye should be parallel to it.

QUIZ QUESTIONS

1. What is the principle of Mohr's method?
2. Name the indicator used in this experiment and what is the end point
3. Can tap water be used for the preparation of AgNO3 solution and why a bottle containing $AgNO_3$ is dark brown or always wrapped with carbon paper?
4. Name the sources of chlorides in water

EXPERIMENT NO:- 5

OBJECT: To determine the iron content in the given sample by redox titration using external indicator.

APPARATUS: Burette, Pipette, conical flask, glass rod, beaker, measuring cylinder.

CHEMICALS: Mohr's salt, N/20 $K_2Cr_2O_7$, Potassium ferricyanide indicator, dilute sulphuric acid

THEORY: Potassium dichromate in acid medium oxidizes ferrous sulphate present in Mohr's salt into ferric sulphate .In this titration, potassium ferricyanide is used as an external indicator which gives a blue colour due to the formation of ferro ferricyanide.

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

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

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

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

(Ferro ferricyanide)
(Blue complex)

PROCEDURE: Pipette out 10 ml of the sample solution in a conical flask and add 5 ml of dilute H_2SO_4. Titrate the solution against N/20 $K_2Cr_2O_7$ solution, using potassium ferricyanide as an external indicator. Take 1 drop of the solution from the conical flask and put it over a drop of potassium ferricyanide solution placed on a white glazed tile. If a blue colour appears, then the end point has not reached. Add more $K_2Cr_2O_7$ solution till a drop of solution does not changed to blue. This is the end point. Repeat till three concordant readings are obtained.

OBSERVATION:

Normality of $K_2Cr_2O_7$solution = N/20

1. Indicator used — Freshly prepared Potassium ferricyanide solution
   End point: Yellow → Brown

OBSERVATION TABLE:

S.No	Volume of Mohr's salt solution in the Titration Flask(ml)	Burette Reading(ml)		Volume of N/20 $K_2Cr_2O_7$ Solution used from burette(ml)
		Initial	Final	
1				
2				
3				

Volume of N/20 $K_2Cr_2O_7$ Solution used from burette (ml) to neutralize10 ml of Mohr's salt solution = $v_2$ml

CALCULATION:

$$N_1V_1 = N_2V_2$$

(Mohr salt sample) ($K_2Cr_2O_7$ solution)

$$N_1 = (1/20 \times v_2\text{ml})/10\text{ml}$$

Strength in terms of Mohr's salt = $N_1 \times 392$ gm/litre

392gm of Mohr's salt contains =56gm of iron

N_1x392 gm of Mohr's salt contains = 56/392× (N_1x392)gm of iron
= $56N_1$ gm of iron
= $56 \times N_1 \times 1000$mgm of iron
= $56 \times N_1 \times 1000$ppm of iron

RESULT: The iron content in the given sample of iron is……ppm.

PRECAUTONS:

1. In each titration same amount of indicator and dilute sulphuric acid should be added
2. There should not be any leakage from burette
3. While taking reading from burette, eye should be parallel to it.

QUIZ QUESTIONS

1. Why is dilute H_2SO_4 and not HCl/HNO_3 used in redox titrations ?
2. What is the formula of ferrous ammonium sulphate and any other name for it?
3. Why is dilute H_2SO_4 added while preparing standard solution of ferrous ammonium sulphate ?

EXPERIMENT NO:- 6

OBJECT: Determination of concentration of HCl solution by titrating it against standard NaOH solution using a pH-meter.

CHEMICALS: HCl, N/10 NaOH, Solutions of pH = 4 & 9

APPARATUS: pH meter, burette, pipette, beakers, measuring cylinder

THEORY: When an alkali is added to an acid solution, the pH of the solution increases slowly, but at the vicinity of the equivalence point, the rate of change of pH of the solution is very rapid. From the sharp break in the curve, we can find the equivalence point, from which the strength can be calculated by normality equation.

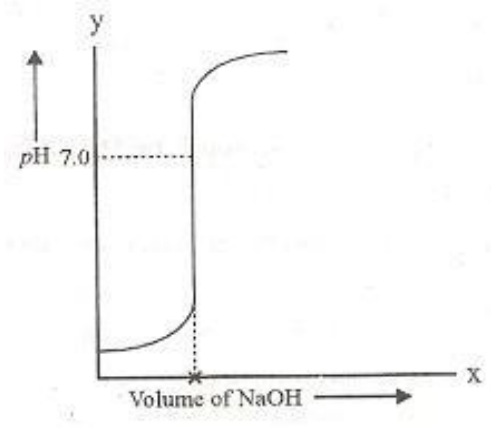
PROCEDURE: First standardize the pH meter with a buffer of pH 4 & 9.Take 50ml of HCl solution in a 100 ml. beaker so that the glass electrodes is completely dipped. Note the pH of pure acid solution. Now add 2 ml of N/10 NaOH from the burette in the beaker. Stir the contents well. Note the pH of the solution. Now go on adding NaOH solution from the burette and note the pH of the solution after each addition of 2 ml of N/10 NaOH. After equivalence point there will be sudden change in pH value i.e it will jump to basic range. After completion of experiment draw a graph between volumes of NaOH added vs pH values. Join two curves and find the volume of N/10 NaOH required to neutralize at pH=7 which is equivalence point

OBSERVATION:

Volume of HCl taken = 50ml
Normality of NaOH solution=N/10

SNO	Vol. of N/10 NaOH	pH	SNO	Vol. of N/10 NaOH	pH
1	0.0		14	26.0	
2	2.0		15	28.0	
3	4.0		16	30.0	
4	6.0		17	32.0	
5	8.0		18	34.0	
6	10.0		19	36.0	
7	12.0		20	38.0	
8	14.0		21	40.0	
9	16.0		22	42.0	
10	18.0		23	44.0	
11	20.0		24	46.0	
12	22.0		25	48.0	
13	24.0		26	50.0	

GRAPH:



Plot between *p*H and volume of NaOH

Volume of N/10 NaOH Solution used from burette (ml) to neutralize50 ml of HCl solution (From Graph) =$v_2$ml

CALCULATIONS:

$$N_1V_1 = N_2V_2$$

($_{HCl\ sample}$) ($_{NaOH\ solution}$)

$$N_1 = (1/10 x v_2 ml)/50ml$$

Strength HCl solution = N_1x36.5 gm/litre

$$= N_1 \times 36.5 \times 1000 mgm/litre$$

$$= N_1 \times 36.5 \times 1000 ppm$$

RESULT: The strength of the given HCl solution = ...ppm.

PRECAUTIONS:

1. pH meter should switched on 15 minutes before performing the experiment
2. pH meter should be standardized properly
3. Temperature should be kept at 25^oC
4. Each time equal amount of NaOH should be added

QUIZ QUESTIONS

1. Define pH and mention the pH of distilled water at 25^0C
2. Do you use any indicator in pH titration?
3. How is the end point determined in pH meteric titration
4. Why is there a sudden jump from acidic value to basic value?

EXPERIMENT NO:- 7

OBJECT: Determination of viscosity of kerosine by Red Wood Viscometer

CHEMICALS: Kerosene oil

THEORY: Viscosity also known as coefficient of viscosity is the tangential force per unit area required to move one horizontal plane of the fluid at unit velocity with respect to another maintained at a unit distance apart by the fluid. The units of viscosity are poise or centipoise. The viscosity of lubricating oil is determined by measuring the time taken for a given quantity of oil to flow through an orifice/hole of standard dimensions.

PROCEDURE: Level the viscometer with the help of leveling screws. Fill the outer bath with water for determining the viscosity at different temperatures. Place the ball valve on the jet to close it. Pour the given sample oil into the cup up to the tip of indicator. Place a clean and dry 100ml calibrated beaker immediately below and directly in line with discharging jet. Insert a thermometer and a stirrer in the cup and cover it with lid. Heat the water filled in the bath slowly with constant stirring. When the oil in the cup attains a desired temperature stop the heating. Lift the ball valve and start the stop watch. Oil from the jet flows into the beaker. Stop the stop watch when the lower meniscus of the oil reaches 50ml mark. Replace the ball valve in the original position to stop the over flow of oil. Record the time taken for 50 ml of oil to collect in the beaker. Repeat the experiment at different temperatures.

OBSERVATION TABLE:

S. No.	Temperature 0C	Viscosity, t sec
1	30	
2	35	
3	40	

RESULT: Viscosity of the oil sample with the help of Redwood Viscometer at …….^{0}C is ……. Redwood sec.

PRECAUTIONS:

1. The oil should be filtered through a fine cloth to remove all solid particles that may clog the jet.
2. The receiving beaker should be place in such a manner that oil does not splash on slab.
3. After each reading the oil should be completely drained from the beaker.

QUIZ QUESTIONS:

1. What do you mean by viscosity?
2. What is the significance of finding the viscosity?
3. What is the effect of temperature on viscosity?
4. How many types of viscometers are there and what is the difference between them?

EXPERIMENT NO: 8

OBJECT: Verification of Beer's law: Determination of iron concentration in sample of water by colorimetry using KCNS as colour developing agent.

APPARATUS: Colorimeter, Burette, Pipette, Measuring Cylinder and Beaker, test tubes, Cuvette

CHEMICALS: 0.1% Fe^{3+} -ion solution (with excess HCl), 20% KCNS in distilled water.

THEORY: Spectroscopy is that branch of science which deals with the interaction of matter with electromagnetic radiations, which are different waves of energy. It covers a wide range of wavelengths or energies and visible light is part of electromagnetic radiations. When monochromatic visible light falls on the homogeneous medium, the intensity of transmitted light is less than that of incident light. A part of incident light has been absorbed.

$$I_o = I_a + I_t$$

I_o- intensity of incident light, I_a- intensity of absorbed light, I_t- intensity of transmitted light

A Spectrometer is a device which detects the percentage transmittance of light radiation, when the light of certain intensity and frequency range is passed through the sample. It is based on Lambert Beer Law which states that "when a monochromatic light is passed through the sample medium, the intensity of that light decreases exponentially with the increase in concentration and thickness of absorbing medium."

$$I_t = I_o e^{-ct}$$

Absorbance or optical density is defined as:

$$A = \log I_o/I_t = €CT$$

A- Absorbance

€- Molar Extinction coefficient or Molar absorptivity

T- Thickness of medium

C- Concentration of the absorbing medium

If the same sample cell i.e. T is constant is used for measurement of absorbance of solution having different concentrations then extent of absorbance (A) is directly proportional to the concentration (C) of the medium.

Thus, if a graph is plotted between A and C, we get a straight line in compliance with Lambert Beer Law. This is known as calibration curve.

For measuring the concentration of given solute, the calibration curve is obtained by measuring the absorbance of standard solution of different concentration. This calibration curve is then used to measure the conc, of unknown solution.

PROCEDURE:

a) **Setting of colorimeter**:
Insert a cuvette containing distilled water in the sample holder adjust the control knob to get zero absorbance

b) **Determination of λ_{max}**:
Insert the cuvette containing given colored solution and note the absorbance reading at different wavelengths or filters. At a specific wavelength maximum absorbance will be observed with given coloured solution

c) **Determination of concentration of sample solution**:
Once λ_{max} is fixed. Prepare the following solution from given sample solution and distilled water using a measuring cylinder

Solution	I	II	III	IV	V	VI	VII	VIII	IX	X

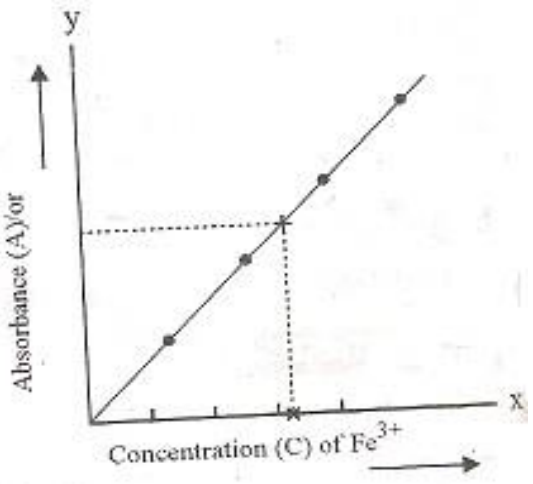
Given sample(ml)	1	2	3	4	5	6	7	8	9	10
Distilled water(ml)	9	8	7	6	5	4	3	2	1	0
% concentration	10	20	30	40	50	60	70	80	90	100

Note down the value of absorbance of above prepared solutions in series. Plot a graph of values of absorbance so obtained and respective concentration should be a straight line. Now note down the absorbance of unknown concentration solution.

OBSERVATION TABLE:

Solution. No.	% Conc. of given sample solution	Absorbance at λ_{max}
I	10	
II	20	
III	30	
IV	40	
V	50	
VI	60	
VII	70	
VIII	80	
IX	90	
X	100	
Unknown solution		

GRAPH:



CALCULATIONS: Concentration of unknown solution from graph= A gm/litre

392 gm of given sample contains=56gm of iron

A gm of given sample contains=56/392×Agm/litre

= (56xA×1000)/392mgm/litre

RESULT: The concentration of Fe (III) in the given sample is obtained from the graph & calculated to be.....ppm

PRECAUTIONS:

1) For the preparation of calibration curve, dilute solutions of unknown concentration should be used
2) λ_{max} should be carefully observed
3) Cuvette should be handled carefully as it is fragile
4) Cuvette should be clean and dry from outside
5) Colorimeter should be switched on 15 minutes before the start of experiment

QUIZ QUESTIONS

1. Explain the law on which this experiment is based and how do we prove it?
2. What is the advantage of colorimeteric method?
3. What is the source of light in colorimeter?
4. Filters are invariable used in absorption spectroscopy? Why?

EXPERIMENT NO:-9

OBJECT: To determine the surface tension of the given liquid by drop number method

APPARATUS: Stalagmometer,a clamp stand, a small rubber tubing with a screw pinch cock,beakers etc.

CHEMICALS: Unknown liquid, water

THEORY: The tension or force in newton acting at right angles along the surface of a liquid one meter in length, is known as surface tension.
Units: dynes/cm in CGS or N/m in SI system.
This method is based on the principal that when a liquid is allowed to pass through a capillary tube, held vertically, at such a slow speed that the drops fall of the tip of the capillary under their won weight and are not pushed away by the kinetic force of flow, the weight of a drop is approximately proportional to the surface tension of the liquid.
The surface tension of a given liquid can be calculated by using the equation

$$y_2 = n_1 p_2 y_1 / n_2 p_1$$

y_1= surface tension of water

y_2= surface tension of water

n_1 = no.of drops of water

n_2 = no. of drops of given of liquid

p_1 = density of water

p_2= density of given liquid

PROCEDURE

1. Thoroughly clean the stalagmometer with chromic acid solution.
2. Rinse it several times with distilled water and finally with alcohol.
3. To the top of stalagmometer, attach the rubber tubing with the screw-pich-cock.
4. Fill the stalagmometer, with distilled water by sucking it above the mark A.
5. Clock the screw- pinch cock.
6. Hold the stalagmometer vertical with the help of a clamp.
7. Slightly open the screw pinch cock and count the number of drops as the liquid falls from the mark A to the mark B.
8. Repeat the process to record at least three numbers of observations.
9. Now, remove the stalagmometer from the stand, wash it thoroughly and dry it.
10. Fill the stalagmometer with the unknown liquid and count the number of drops and count the number of drops as with water.
11. Repeat it thrice.

OBERVATION

Density of water , p_1 =

Density of unknown liquid, p_2 =

Surface tension of water, y_1 = 72.14dynes/cm

S No	Water		Given liquid	
	No. of drops	Mean (n1)	No. of drops	mean (n2)
1				
2				
3				

CALCULATIONS:

Surface tension of given liquid (y_2) = $n_1p_2y_1/n_2p_1$

RESULT :

Surface tension of the given liquid = (dynes/cm)

PRECAUTIONS:

1. Before use, the stalgmometr should be thoroughly cleaned
2. While closing the screw -pinch-cock, care should be taken that the liquid meniscus does not fall below the mark A.
3. The number of drops per minute should not exceed 15 otherwise they may not be properly formed.
4. While counting the drops, all disturbances of the stalgmometer should be avoided.
5. As far as possible, the temperature should remain constant.
6. At least three observations for water and liquid should be recorded.

QUIZ QUESTIONS

1. What is surface tension?
2. Name the apparatus used for the determination of surface tension of a liquid
3. How the surface tension of a liquid is related to temperature?

EXPERIMENT NO :10

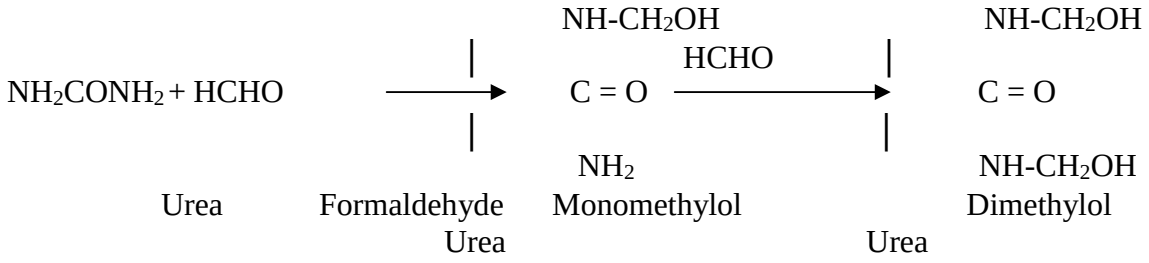
10 (A):-Preparation of Urea formaldehyde and Phenol formaldehyde

AIM: To prepare Urea formaldehyde resin

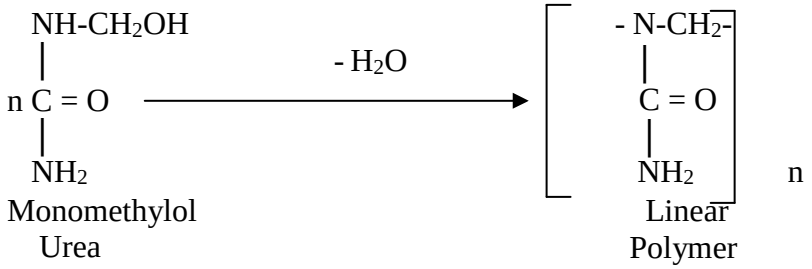
APPARATUS: Beaker, glass rod, filter paper and funnel

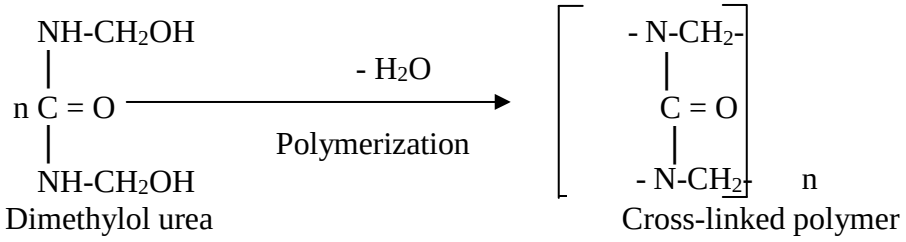
CHEMICALS: Urea, formaldehyde solution, conc. Sulphuric acid and distilled water

THEORY: Urea formaldehyde is prepared by condensation reaction between urea and formaldehyde in acidic or alkaline medium. The first product formed during the reaction is monomethylol and dimethylol ureas.

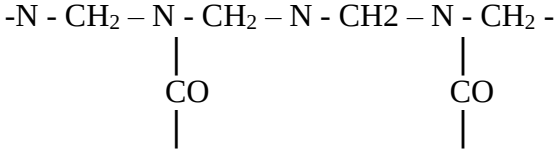


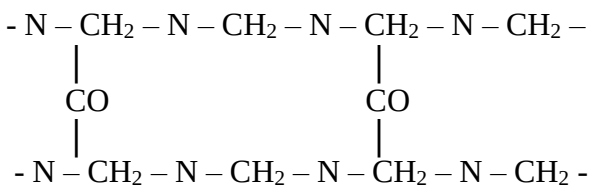
Polymerization can take place from mono or dimethylol urea or possibly through both, with the formation of long chains.





A fully cross-linked urea formaldehyde resin can be represented as:-





Urea formaldehyde resin (cross-linked polymer)

OBSERVATIONS: -

Mass of the beaker (W1) = ---------g.
Mass of the beaker with urea formaldehyde (W2) = --------g.
Therefore mass of urea formaldehyde (W2 – W1) = --------g.

RESULT: - The yield of urea formaldehyde = -------g

PRECAUTIONS:-

1. While adding concentrated H_2SO4, it is better to stay little away from the beaker since the reaction sometimes becomes vigorous.
2. The reaction mixture should be stirred continuously.

10 (B):-

OBJECT: To prepare phenol formaldehyde (Bakelite) resin

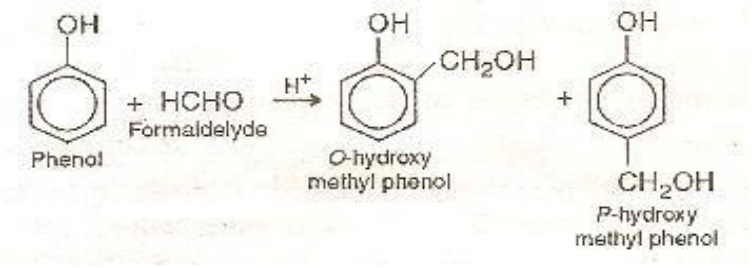
APPARATUS: Measuring cylinder, beakers, glass rod, funnel, filter papers

CHEMICALS: Phenol, Glacial acetic acid, Formaldehyde, and conc. HCl acid

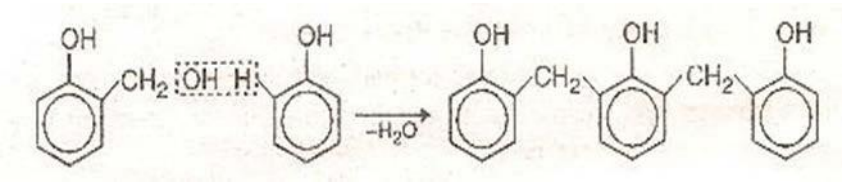
THEORY:

Phenol formaldehyde resin can be prepared by condensation polymerization phenol with formaldehyde in acidic medium. Reaction takes place in three steps;

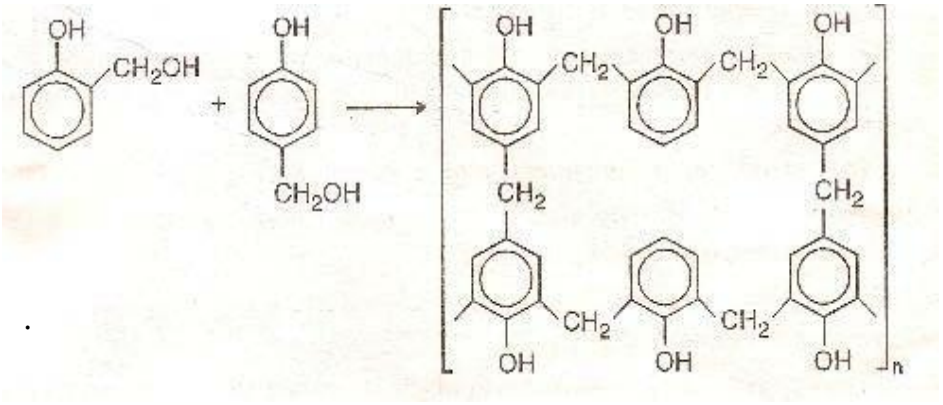
Step1: Phenol reacts with formaldehyde in acidic medium



Step2: Hydroxy methyl phenol reacts with phenol to give linear polymer.



Step3: It polymerizes at high temperature into a three dimensional cross-linked polymer.



PROCEDURE FOR PREPATION OF BAKELITE:

Tak5ml of glacial acetic acid and 2.5ml of 40% formaldehyde solution in a beaker. To this add 2ml of phenol. Add 5ml of Conc. HCl acid to the reaction mixture drop wise with continuous stirring till pink colour mass appears. Residue obtained is washed with water several times till it is free from acid. Filter and dry the product and note its weight.

RESULT: Weight of Phenol formaldehyde =

PRECAUTIONS:

1) Reaction is vigorous. All addition should be done carefully and with continuous stirring.
2) Fuming cupboard should be used for preparation.

QUIZ QUESTIONS

1. Define a polymer
2. What is the common name of phenol formaldehyde resin
3. Define condensation polymerization
4. Write the polymerization reactions for the formation of urea formaldehyde resin.

This lab manual has been updated by

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Crosschecked ByHOD APS

Verified By
Director, DGI Greater Noida

Please spare some time to provide your valuable feedback.