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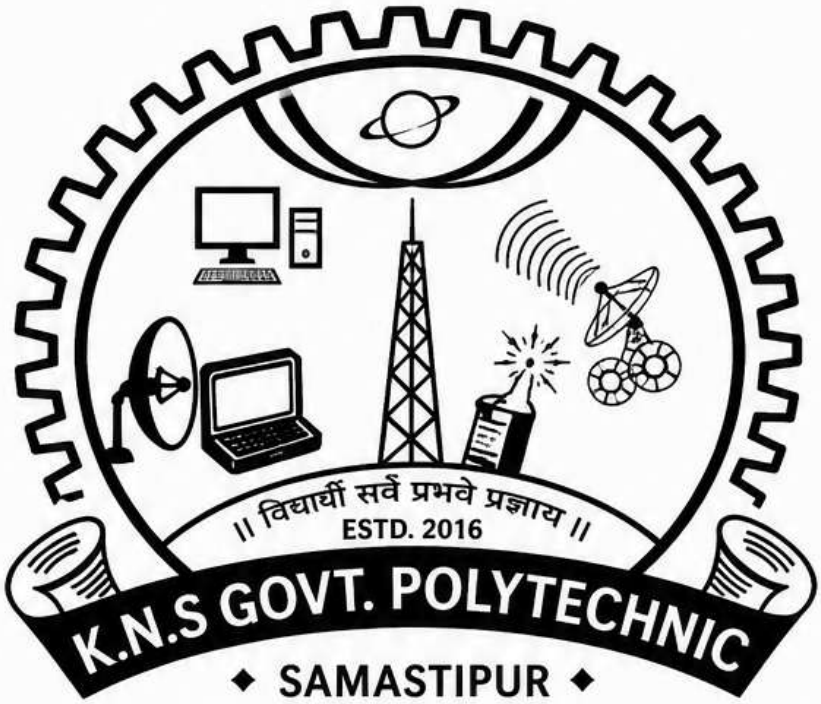

APPLIED CHEMISTRY
PRACTICAL LAB MANUAL

NAME OF STUDENT	:	_____
ROLL NUMBER	:	_____
REGISTRATION NUMBER	:	_____
BRANCH	:	_____
SEMESTER	:	_____
ACADEMIC SESSION	:	_____

CERTIFICATE

This is to certify that Mr./Ms. _____ of _____ has satisfactorily completed the prescribed Chemistry practical work and maintained this laboratory manual during the academic session _____.

Faculty In-charge: _____

Head of Department: _____

ACKNOWLEDGEMENT

I sincerely acknowledge the guidance of my faculty members and laboratory staff during the completion of the Chemistry practical work. This manual has been maintained to record the scientific principles, procedures, observations, calculations and results of the prescribed experiments.

GENERAL LABORATORY INSTRUCTIONS

1. Read the complete procedure before starting the experiment.
2. Wear appropriate laboratory coat and protective equipment where required.
3. Never pipette by mouth and never taste chemicals.
4. Use clean glassware and label prepared solutions.
5. Report spills, breakage and accidents immediately.
6. Record observations at the time of the experiment.
7. Dispose of chemical waste only as directed by the laboratory staff.

TABLE OF EXPERIMENTS

No.	Experiment	Date	Faculty Sign.
01	Preparation of 250 mL N/10 Oxalic Acid Solution		
02	Preparation of 250 mL N/10 Sodium Carbonate Solution		
03	Determination of Strength of Sodium Hydroxide Using Standard Oxalic Acid		
04	Determination of Total Hardness of Water by EDTA Method		
05	Estimation of Moisture Content in Coal		
06	Preparation of Barium Sulphate from Barium Chloride		
07	Determination of Viscosity Using Ostwald Viscometer		
08	Effect of Dilution on EMF of a Daniell Cell		
09	Determination of pH Using a pH Meter		
10	Preparation of Phenol-Formaldehyde Resin (Bakelite)		
11	Determination of Dissolved Oxygen by Winkler Method		
12	Qualitative Study of Soil pH Nature Using Vinegar and Baking Soda		

EXPERIMENT NO. 01

AIM

To prepare 250 mL of accurately known N/10 oxalic acid solution.

THEORY

A standard solution is a solution whose concentration is known accurately. Oxalic acid dihydrate is commonly used as a primary standard because it is available in a sufficiently pure and stable form. The required mass is calculated from normality, equivalent weight and the desired volume.

PRINCIPLE

Mass required (g) = Normality x Equivalent weight x Volume (L). For oxalic acid dihydrate, equivalent weight = 63. Therefore, for 250 mL of N/10 solution: 0.1 x 63 x 0.250 = 1.575 g.

CHEMICAL REACTION / SCIENTIFIC BASIS

The prepared oxalic acid solution can later act as a standard acid in acid-base titrations.

APPARATUS / CHEMICALS REQUIRED

Oxalic acid dihydrate, distilled water, analytical balance, watch glass, beaker, glass rod, funnel and 250 mL volumetric flask.

PROCEDURE

1. Accurately weigh 1.575 g of oxalic acid dihydrate.
2. Dissolve it completely in about 100-150 mL of distilled water.
3. Transfer the solution quantitatively into a 250 mL volumetric flask.
4. Wash the beaker, glass rod and funnel with distilled water and add all washings.
5. Make the final volume exactly up to the calibration mark.
6. Stopper and invert the flask several times to obtain a uniform solution.

OBSERVATION TABLE

Observation	Value / Reading	Unit	Remarks
Mass of oxalic acid		g	
Final volume		mL	
Calculated normality		N	

CALCULATION

Mass required = N x Eq. wt. x V(L) = 0.1 x 63 x 0.250 = 1.575 g.

Working Space	

RESULT

250 mL of N/10 oxalic acid solution is prepared.

PRECAUTIONS	SOURCES OF ERROR
- Use clean glassware and distilled water.	- Incorrect weighing.
- Do not exceed the calibration mark.	- Loss of solution during transfer.
- Ensure complete quantitative transfer of the solute.	- Incorrect final meniscus reading.

VIVA QUESTIONS

1. Why is oxalic acid used as a primary standard?

Ans. Because a sufficiently pure and stable substance can be weighed directly to prepare a solution of accurately known concentration.

2. Why are washings added to the volumetric flask?

Ans. To ensure complete quantitative transfer of the solute.

EXPERIMENT NO. 02

AIM

To prepare 250 mL of N/10 sodium carbonate solution.

THEORY

Normality expresses the number of gram-equivalents of solute present per litre of solution. Anhydrous sodium carbonate reacts quantitatively with acids and is used in acid-base chemistry. Accurate weighing and dilution to a fixed volume are essential for correct concentration.

PRINCIPLE

For sodium carbonate, equivalent weight = molecular mass / 2 = 53. Required mass = 0.1 x 53 x 0.250 = 1.325 g.

CHEMICAL REACTION / SCIENTIFIC BASIS

$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2(g)$.

APPARATUS / CHEMICALS REQUIRED

Anhydrous sodium carbonate, distilled water, balance, beaker, glass rod, funnel and 250 mL volumetric flask.

PROCEDURE

1. Accurately weigh 1.325 g of dry anhydrous sodium carbonate.
2. Dissolve it completely in distilled water.
3. Transfer the solution quantitatively to a 250 mL volumetric flask.
4. Wash all transfer apparatus into the flask.
5. Make the volume exactly up to the mark.
6. Stopper and invert several times to mix thoroughly.

OBSERVATION TABLE

Observation	Value / Reading	Unit	Remarks
Mass of Na_2CO_3		g	
Final volume		mL	
Calculated normality		N	

CALCULATION

Mass required = N x Eq. wt. x V(L) = 0.1 x 53 x 0.250 = 1.325 g.

Working Space	

RESULT

250 mL of N/10 sodium carbonate solution is prepared.

PRECAUTIONS	SOURCES OF ERROR
- Use dry anhydrous sodium carbonate.	- Moisture in the reagent.
- Avoid loss during transfer.	- Incorrect weighing.
- Read the bottom of the meniscus at eye level.	- Incorrect final volume.

VIVA QUESTIONS

1. Why is the anhydrous form specified?

Ans. Water associated with the reagent changes the mass required for the desired concentration.

2. Why is a volumetric flask used?

Ans. It is calibrated to contain an accurate fixed volume.

EXPERIMENT NO. 03

AIM

To determine the normality and strength of the given sodium hydroxide solution by acid-base titration.

THEORY

Acid-base titration is based on neutralisation. A standard solution of oxalic acid reacts quantitatively with sodium hydroxide. Phenolphthalein is suitable because its colour change occurs in the alkaline pH range. The end point is the first faint pink colour that persists for a short time.

PRINCIPLE

At equivalence, acid equivalents = base equivalents. Therefore, N1V1 = N2V2. Strength of sodium hydroxide in g/L = Normality x equivalent weight, where the equivalent weight of NaOH is 40.

CHEMICAL REACTION / SCIENTIFIC BASIS

H2C2O4 + 2NaOH -> Na2C2O4 + 2H2O.

APPARATUS / CHEMICALS REQUIRED

N/10 oxalic acid, given NaOH solution, phenolphthalein, burette, pipette, conical flask and white tile.

PROCEDURE

1. Rinse and fill the burette with the given NaOH solution and remove air bubbles.
2. Pipette 10 mL of standard oxalic acid into a conical flask.
3. Add 2-3 drops of phenolphthalein indicator.
4. Titrate with NaOH while swirling the flask.
5. Near the end point, add NaOH dropwise.
6. Stop at the first faint pink colour persisting for about 30 seconds.
7. Repeat until concordant readings are obtained.

OBSERVATION TABLE

Trial	Initial burette	Final burette	Titre / mL	Remarks
I				
II				
III				

CALCULATION

N(acid) x V(acid) = N(base) x V(base). Strength of NaOH = N(base) x 40 g/L.

Working Space	

RESULT

The normality and strength of the given NaOH solution are calculated from concordant titre values.

PRECAUTIONS	SOURCES OF ERROR
- Remove air bubbles from the burette tip.	- Parallax in burette reading.
- Do not overshoot the faint-pink end point.	- Overshooting the end point.
- Use concordant readings for the final calculation.	- Incorrect indicator quantity.

VIVA QUESTIONS

1. Why is a white tile used?

Ans. It makes the colour change at the end point easier to observe.

2. Why is phenolphthalein suitable?

Ans. Its transition range is suitable for the end point of this titration.

EXPERIMENT NO. 04

Determination of Total Hardness of Water by EDTA Method

Date of Experiment	: _____
Faculty Signature	: _____

AIM

To determine the total hardness of the given water sample by complexometric titration using standard EDTA solution.

THEORY

Hardness of water is mainly caused by dissolved salts of calcium and magnesium. In complexometric titration, EDTA forms stable 1:1 chelate complexes with Ca^{2+} and Mg^{2+}. At about pH 10, Eriochrome Black T forms a wine-red complex with the metal ions. During titration, EDTA binds the metal ions more strongly and the indicator changes to blue at the end point.

PRINCIPLE

The amount of standard EDTA required is directly proportional to the total amount of calcium and magnesium ions present in the measured water sample. The hardness is conventionally expressed as mg/L (or ppm) as $CaCO_3$.

CHEMICAL REACTION / SCIENTIFIC BASIS

$M^{2+} + EDTA^{4-} \rightarrow M\text{–}EDTA^{2-}$ (M = Ca or Mg; simplified representation)

APPARATUS / CHEMICALS REQUIRED

Apparatus: Burette, pipette, conical flask and measuring cylinder.
Chemicals: Water sample, standard EDTA solution, pH 10 buffer and Eriochrome Black T indicator.

PROCEDURE

1. Fill the burette with standardised EDTA solution and remove all air bubbles from the tip.
2. Pipette an accurately measured volume of the water sample into a clean conical flask.
3. Add the prescribed quantity of pH 10 buffer solution.
4. Add a small amount of Eriochrome Black T indicator; the solution should become wine-red.
5. Titrate with EDTA while continuously swirling the flask.
6. Near the end point, add EDTA slowly until the colour changes sharply from wine-red to clear blue.
7. Repeat the titration until concordant readings are obtained and use the average titre for calculation.

OBSERVATION TABLE

Trial	Initial Reading	Final Reading	EDTA Used (mL)	Remarks
1				
2				
3				

CALCULATION

Method A, when EDTA factor is known: Total hardness (mg/L as CaCO3) = (V_EDTA x F x 1000) / V_sample. Method B, when EDTA normality is known: Total hardness (mg/L as CaCO3) = (V_EDTA x N_EDTA x 50,000) / V_sample.

RESULT

Total hardness of the given water sample = _____ mg/L as CaCO3.

PRECAUTIONS

- Maintain the required pH near 10.
- Use standardised EDTA and a clean burette.
- Use the same accurately measured sample volume for each trial.
- Observe the wine-red to blue end point carefully.

SOURCES OF ERROR

- Incorrect EDTA factor or normality.
- Incorrect pH.
- Poor end-point judgement.
- Parallax error in burette readings.

VIVA QUESTIONS

1. **What causes water hardness?**

Ans. Mainly dissolved calcium and magnesium salts.

2. **Why is buffer added?**

Ans. To maintain the pH required for stable complex formation and a sharp indicator end point.

3. **Why is hardness expressed as CaCO3?**

Ans. CaCO3 provides a common standard for comparing total hardness.

4. **What is the EDTA to metal-ion ratio?**

Ans. For Ca2+ and Mg2+, EDTA forms complexes in a 1:1 molar ratio.

EXPERIMENT NO. 05

AIM

To determine the percentage moisture present in the given coal sample.

THEORY

Moisture is the water associated with coal. On heating under controlled conditions, removable moisture is lost, causing a decrease in mass. The difference between the initial mass and the dried constant mass is used to calculate moisture percentage.

PRINCIPLE

Under the prescribed heating conditions, loss in mass is attributed to moisture. Repeated heating and weighing until constant mass improves reliability.

CHEMICAL REACTION / SCIENTIFIC BASIS

This is a gravimetric loss-on-drying determination; no specific chemical reaction is required.

APPARATUS / CHEMICALS REQUIRED

Coal sample, clean crucible or weighing dish, hot-air oven, desiccator, balance and tongs.

PROCEDURE

1. Weigh a clean, dry dish.
2. Add a known mass of powdered coal and weigh again.
3. Heat at the prescribed temperature for the specified time.
4. Cool the sample in a desiccator.
5. Weigh the cooled sample.
6. Repeat heating, cooling and weighing until constant mass is obtained.
7. Calculate percentage moisture from the mass loss.

OBSERVATION TABLE

Observation	Mass / g	Calculation	Remarks
Empty dish			
Dish + coal before heating			
Dish + coal after heating			

CALCULATION

Percentage moisture = (Loss in mass / Initial mass of coal) x 100.

Working Space	

RESULT

The percentage moisture content of the given coal sample is calculated.

PRECAUTIONS	SOURCES OF ERROR
- Do not weigh a hot sample.	- Sample absorbing moisture during cooling.
- Cool in a desiccator before weighing.	- Incomplete drying.
- Use a clean, dry weighing container.	- Loss of sample during handling.

VIVA QUESTIONS

1. Why is a desiccator used?

Ans. To prevent the dried sample from absorbing moisture from air.

2. Why is constant mass important?

Ans. It indicates that further drying no longer produces a significant mass change.

EXPERIMENT NO. 06

AIM

To prepare barium sulphate by a precipitation reaction.

THEORY

When soluble barium ions react with sulphate ions, the sparingly soluble salt barium sulphate forms as a white precipitate. Filtration separates the insoluble product from soluble materials, and washing removes adhering impurities.

PRINCIPLE

The experiment is based on a double-displacement precipitation reaction and the very low solubility of barium sulphate.

CHEMICAL REACTION / SCIENTIFIC BASIS

$BaCl_2 + H_2SO_4 \rightarrow BaSO_4(s) + 2HCl$.

APPARATUS / CHEMICALS REQUIRED

Barium chloride solution, prescribed sulphate reagent, beaker, glass rod, funnel, filter paper and wash bottle.

PROCEDURE

1. Take the prescribed barium chloride solution in a clean beaker.
2. Add the sulphate reagent slowly with continuous stirring.
3. Allow complete formation and settling of the white precipitate.
4. Filter through properly fitted filter paper.
5. Wash the precipitate with distilled water.
6. Dry the product if required by the laboratory procedure.

OBSERVATION TABLE

Observation	Reading / Description	Unit	Remarks
Appearance of precipitate			
Mass of dry product		g	
Percentage yield, if required		%	

CALCULATION

Reaction-based preparation. If product yield is required: Percentage yield = (actual yield / theoretical yield) x 100.

Working Space	

RESULT

White barium sulphate is prepared.

PRECAUTIONS	*SOURCES OF ERROR*

- Handle barium-containing chemicals carefully.	- Incomplete precipitation.
- Add reagents slowly to avoid splashing.	- Loss of precipitate during transfer.
- Wash the precipitate thoroughly as directed.	- Contamination during filtration.

VIVA QUESTIONS

1. Why is washing the precipitate necessary?

Ans. To remove soluble impurities and adhering reagents.

2. What type of reaction is involved?

Ans. A precipitation or double-displacement reaction.

EXPERIMENT NO. 07

AIM

To determine the viscosity or relative viscosity of a given liquid using an Ostwald viscometer.

THEORY

Viscosity is the resistance offered by a fluid to flow. In an Ostwald viscometer, a fixed volume of liquid flows through a capillary under gravity. Under comparable conditions, viscosity is related to liquid density and the measured flow time between two marks.

PRINCIPLE

For two liquids measured under comparable conditions: eta1 / eta2 = (rho1 x t1) / (rho2 x t2).

CHEMICAL REACTION / SCIENTIFIC BASIS

No chemical reaction is involved; this is a physical-property measurement based on capillary flow.

APPARATUS / CHEMICALS REQUIRED

Ostwald viscometer, distilled water, given liquid, stopwatch, thermometer and stand.

PROCEDURE

1. Clean the viscometer and mount it vertically.
2. Measure the flow time of distilled water between the timing marks.
3. Repeat and obtain an average time.
4. Repeat the measurement for the given liquid at the same temperature.
5. Record density values if required.
6. Use the density and flow-time relation for calculation.

OBSERVATION TABLE

Liquid	Density	Time 1 / s	Time 2 / s	Average / s
Water				
Given liquid				
Temperature			deg C	

CALCULATION

eta(sample) / eta(reference) = [rho(sample) x t(sample)] / [rho(reference) x t(reference)].

Working Space	

RESULT

The viscosity or relative viscosity of the given liquid is determined.

PRECAUTIONS	SOURCES OF ERROR
- Maintain constant temperature.	- Temperature variation.
- Keep the viscometer vertical.	- Stopwatch reaction-time error.
- Ensure there are no air bubbles in the capillary.	- Improper vertical alignment.

VIVA QUESTIONS

1. Why is temperature important?

Ans. Viscosity changes significantly with temperature.

2. Why is the viscometer kept vertical?

Ans. The flow conditions must remain consistent for accurate comparison.

EXPERIMENT NO. 08

Effect of Dilution on EMF of a Daniell Cell

Date of Experiment	: _____
Faculty Signature	: _____

AIM

To study the effect of electrolyte concentration on the EMF of a Daniell cell.

THEORY

A Daniell cell converts chemical energy into electrical energy through a spontaneous redox reaction between zinc and copper ions. The electrode potentials depend on ionic activities; therefore, changing the concentration of a half-cell solution can change the reaction quotient and hence the measured EMF.

PRINCIPLE

The cell is represented as Zn | Zn^{2+} || Cu^{2+} | Cu. The overall reaction is Zn + Cu^{2+} → Zn^{2+} + Cu. At 25°C, the EMF depends on the ratio of ionic activities according to the Nernst equation.

CHEMICAL REACTION / SCIENTIFIC BASIS

$E_{cell} = E°_{cell} - (0.05916/2) \log(a_{Zn2+} / a_{Cu2+})$ at 25°C. For sufficiently dilute solutions, activity may be approximated by concentration.

APPARATUS / CHEMICALS REQUIRED

Apparatus: Zinc electrode, copper electrode, two beakers, salt bridge and high-resistance voltmeter or potentiometer.
Chemicals: Zinc sulphate solution and copper sulphate solution.

PROCEDURE

1. Clean the zinc and copper electrodes and prepare the two half-cells.
2. Connect the half-cells using a properly prepared salt bridge.
3. Connect the cell to a high-resistance voltmeter or potentiometer and record the stable EMF.
4. Dilute the prescribed half-cell solution by the required amount while keeping other conditions unchanged.
5. Allow the reading to stabilise and record the new EMF.
6. Repeat the observation for the required concentrations.
7. Compare the observed change in EMF with the Nernst equation.

OBSERVATION TABLE

Trial	Concentration	Other Half-cell	EMF (V)	Temperature (C)
1				
2				
3				

CALCULATION

At 25 C: Ecell = E0cell - (0.05916/2) log Q, where Q = a_Zn2+ / a_Cu2+. For dilute solutions, Q may be approximated as [Zn2+] / [Cu2+].

RESULT

The effect of the prescribed electrolyte dilution on the EMF of the Daniell cell was observed and recorded.

PRECAUTIONS

- Do not allow direct mixing of the two half-cell solutions.
- Keep electrodes clean and properly immersed.
- Use a stable salt bridge.
- Allow the reading to stabilise before recording.
- Keep temperature as constant as possible.

SOURCES OF ERROR

- Dirty or polarised electrodes.
- Unstable salt bridge or liquid-junction effects.
- Incorrect concentration after dilution.
- Temperature variation.
- Instrument loading or unstable reading.

VIVA QUESTIONS

1. **What is the function of the salt bridge?**

Ans. It completes the circuit and helps maintain electrical neutrality.

2. **Why can dilution change the EMF?**

Ans. Selective dilution changes ionic activities and therefore the reaction quotient in the Nernst equation.

3. **What is the value of n for the Daniell cell?**

Ans. n = 2 electrons are transferred in the overall cell reaction.

4. **What happens if both ionic concentrations are diluted equally?**

Ans. Ideally the concentration ratio remains unchanged, so the Nernst contribution to EMF is essentially unchanged.

EXPERIMENT NO. 09

AIM

To determine the pH of the given solution using a calibrated pH meter.

THEORY

pH is a logarithmic measure related to hydrogen-ion activity. A pH meter uses an electrode system, commonly including a glass electrode and reference electrode, to measure a potential difference that is converted to pH after calibration with standard buffers.

PRINCIPLE

pH = -log10(aH+). Calibration establishes the relationship between electrode response and known buffer values.

CHEMICAL REACTION / SCIENTIFIC BASIS

No single chemical reaction is involved; the measurement is based on electrode potential developed due to hydrogen-ion activity.

APPARATUS / CHEMICALS REQUIRED

pH meter, suitable buffer solutions, electrode, distilled water, sample beaker and tissue paper.

PROCEDURE

1. Switch on the pH meter and allow it to stabilise.
2. Calibrate with prescribed standard buffer solutions.
3. Rinse the electrode with distilled water and gently blot it.
4. Immerse the electrode in the sample without touching the sides or bottom.
5. Wait for a stable reading and record the pH.
6. After measurement, rinse the electrode and store it properly.

OBSERVATION TABLE

Trial	Buffer / Sample	Expected pH	Meter Reading	Remarks
1	Buffer pH 4			
2	Buffer pH 7			
3	Sample solution			

CALCULATION

No calculation is normally required. The direct pH value is obtained from the calibrated instrument.

Working Space	

RESULT

The pH of the given solution is measured and recorded.

PRECAUTIONS	SOURCES OF ERROR
- Calibrate the pH meter before use. - Do not rub the glass bulb. - Do not allow the electrode to dry out.	- Incorrect calibration. - Dirty electrode. - Temperature variation. - Not waiting for a stable reading.

VIVA QUESTIONS

1. What is pH?

Ans. It is the negative logarithm of hydrogen-ion activity.

2. Why are buffer solutions used?

Ans. They provide known pH values for calibration.

EXPERIMENT NO. 10

AIM

To prepare phenol-formaldehyde resin by condensation polymerisation under prescribed laboratory conditions.

THEORY

Phenol reacts with formaldehyde to form polymeric phenol-formaldehyde materials. Depending on reagent ratio and reaction conditions, intermediate products can further cross-link to form a hard thermosetting network commonly associated with Bakelite-type resin.

PRINCIPLE

Repeated condensation reactions form larger molecules. On further curing, extensive cross-linking produces a rigid thermosetting polymer.

CHEMICAL REACTION / SCIENTIFIC BASIS

Phenol + formaldehyde -> hydroxymethyl phenol intermediates -> phenol-formaldehyde resin + condensation by-products.

APPARATUS / CHEMICALS REQUIRED

Phenol, formaldehyde solution, prescribed catalyst, heating arrangement, beaker, stirrer, filtration setup and distilled water.

PROCEDURE

1. Wear appropriate PPE and work under direct supervision.
2. Mix the prescribed quantities of reagents in the specified sequence.
3. Add the prescribed catalyst carefully.
4. Warm gently while stirring for the required duration.
5. Allow resin formation to occur.
6. Wash, separate, filter and dry the product as directed.

OBSERVATION TABLE

Observation	Reading / Description	Unit	Remarks
Initial appearance			
Time of resin formation		min	
Final product appearance			

CALCULATION

Polymer formation depends on stoichiometry and reaction conditions. Use only the quantities specified by the laboratory.

Working Space	

RESULT

Phenol-formaldehyde resin is prepared.

PRECAUTIONS	SOURCES OF ERROR
- Phenol and formaldehyde are hazardous; use PPE and ventilation.	- Incorrect reagent ratio.
- Do not heat aggressively.	- Insufficient mixing.
- Follow the instructor's prescribed quantities and conditions.	- Incorrect heating conditions.

VIVA QUESTIONS

1. Why is Bakelite called a thermosetting polymer?

Ans. On curing, extensive cross-linking forms a rigid network that does not melt like a thermoplastic.

2. What type of polymerisation is involved?

Ans. Condensation polymerisation.

EXPERIMENT NO. 11

Determination of Dissolved Oxygen by Winkler Method

Date of Experiment	: _____
Faculty Signature	: _____

AIM

To determine the dissolved oxygen concentration in the given water sample by the Winkler iodometric method.

THEORY

Dissolved oxygen is molecular oxygen present in water and is an important indicator of water quality. In the Winkler method, oxygen oxidises manganese(II) under alkaline conditions. After acidification, iodine is liberated in an amount chemically equivalent to the dissolved oxygen originally present. The liberated iodine is titrated with standard sodium thiosulphate.

PRINCIPLE

The amount of sodium thiosulphate required to reduce the liberated iodine is stoichiometrically proportional to the dissolved oxygen fixed in the original water sample.

CHEMICAL REACTION / SCIENTIFIC BASIS

$I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$. The iodometric reaction sequence links the amount of iodine liberated to the original dissolved oxygen content.

APPARATUS / CHEMICALS REQUIRED

Apparatus: BOD bottle, burette, pipette, conical flask and measuring cylinder.
Chemicals: Water sample, manganese sulphate reagent, alkaline iodide/azide reagent, sulphuric acid, standard sodium thiosulphate and starch indicator.

PROCEDURE

1. Fill the BOD bottle completely with the water sample without trapping air bubbles.
2. Add the prescribed amount of manganese sulphate reagent followed by alkaline iodide/azide reagent.
3. Stopper immediately, mix thoroughly and allow the precipitate to settle.
4. Add sulphuric acid as prescribed and mix until the precipitate dissolves and iodine is liberated.
5. Take the prescribed aliquot or titrate the specified volume according to the laboratory procedure.
6. Titrate with standard sodium thiosulphate until the solution becomes pale yellow.
7. Add starch indicator and continue titration dropwise until the blue colour just disappears.
8. Repeat if required and use concordant titre values.

OBSERVATION TABLE

Trial	Initial Reading	Final Reading	Titre (mL)	Remarks
1				
2				
3				

CALCULATION

DO (mg/L) = (Vt x Nt x 8000) / Veff, where Vt = mL of sodium thiosulphate used, Nt = normality of sodium thiosulphate, and Veff = effective volume in mL of original sample represented by the titrated portion.

> Important: Use the exact sample-volume convention prescribed by your college laboratory because Winkler procedures can use different bottle volumes and aliquot corrections.

RESULT

Dissolved oxygen in the given water sample = _____ mg/L.

PRECAUTIONS

- Avoid trapping air bubbles in the BOD bottle.
- Fix the sample immediately after collection.
- Use correctly standardised sodium thiosulphate.
- Add starch near the end point, not at the beginning.
- Follow the prescribed sample-volume and aliquot corrections exactly.

SOURCES OF ERROR

- Air contact with the sample.
- Delay before fixation.
- Incorrect thiosulphate normality.
- Incorrect effective sample volume.
- Over-titration of the starch end point.

VIVA QUESTIONS

1. **Why must the BOD bottle be filled without bubbles?**

Ans. Air exchange can change the oxygen content and affect the result.

2. **Why is starch added near the end point?**

Ans. It gives a sensitive blue colour with iodine and works best when iodine concentration is low.

3. **What does the factor 8000 represent?**

Ans. It combines equivalent-mass and litre conversion factors when volumes are expressed in mL.

4. **Why must the effective sample volume be used?**

Ans. The calculation must represent the actual original sample volume corresponding to the titrated iodine.

EXPERIMENT NO. 12

Qualitative Study of Soil Acid-Base Nature Using Vinegar and Baking Soda

Date of Experiment	: _____
Faculty Signature	: _____

AIM

To qualitatively observe the acid-base behaviour of the given soil using simple vinegar and baking-soda tests; exact soil pH, if required, must be measured separately using a calibrated pH method.

THEORY

Simple fizzing tests can indicate certain acid-base reactions, but they do not directly measure pH. Carbonate-rich or calcareous soil can effervesce when vinegar is added because carbon dioxide is released. Baking soda is not a reliable quantitative reagent for determining numerical soil pH; therefore, any reaction observed with it should be recorded only as a qualitative observation.

PRINCIPLE

Carbonate or bicarbonate present in soil reacts with acid to produce carbon dioxide gas. Exact soil pH requires a calibrated pH meter or another prescribed standard soil-water or soil-salt suspension method.

CHEMICAL REACTION / SCIENTIFIC BASIS

$CO_3^{2-} + 2H^+ \rightarrow CO_2(g) + H_2O$ and $HCO_3^- + H^+ \rightarrow CO_2(g) + H_2O$

APPARATUS / CHEMICALS REQUIRED

Apparatus: Clean containers, measuring spoon, dropper and, for exact pH, a calibrated pH meter.
Chemicals / Materials: Representative soil sample, distilled water, vinegar and baking soda.

PROCEDURE

1. Place equal amounts of representative soil in clean, labelled containers.
2. Add a small quantity of distilled water if a comparable soil slurry is required by the laboratory procedure.
3. Add vinegar to one portion and observe whether sustained effervescence occurs; this may indicate carbonate or bicarbonate content.
4. If baking soda is used as prescribed by the syllabus, record only the visible qualitative behaviour and do not convert it into a numerical pH value.
5. For exact soil pH, prepare the prescribed soil suspension and measure it with a calibrated pH meter.
6. Record all observations clearly and state whether the conclusion is qualitative or based on an actual pH measurement.

OBSERVATION TABLE

Test	Reagent / Method	Observation	Interpretation	Remarks
A	Vinegar		Carbonate indication, if any	
B	Baking soda		Qualitative only	
C	Calibrated pH method		Record actual pH	

CALCULATION

No numerical pH is calculated from fizzing alone. If a pH meter is used, report the direct calibrated meter reading and state the soil-to-liquid ratio used in sample preparation.

> Scientific note: The vinegar and baking-soda tests are qualitative acid-base or carbonate observations and are not a substitute for an actual soil pH determination.

RESULT

The soil acid-base behaviour was qualitatively observed. Exact pH, when required, must be reported from a calibrated pH measurement: pH = _____.

PRECAUTIONS

- Use representative and uncontaminated soil.
- Use equal sample quantities for comparison.
- Use clean containers and distilled water.
- Do not treat fizzing intensity as an exact pH value.
- Calibrate the pH meter before an exact measurement.

SOURCES OF ERROR

- Unequal sample quantities.
- Subjective observation of fizzing.
- Contamination by previous samples.
- Incorrect soil-water ratio for pH measurement.
- Uncalibrated or poorly maintained pH electrode.

VIVA QUESTIONS

1. **Why is the vinegar test qualitative?**

Ans. Effervescence only indicates a possible acid-carbonate reaction and does not provide a calibrated numerical pH.

2. **What does strong effervescence with vinegar usually suggest?**

Ans. It suggests the presence of appreciable carbonate or bicarbonate material.

3. **Can baking soda alone determine soil pH?**

Ans. No. It is not a calibrated quantitative method for determining numerical soil pH.

4. **How should exact soil pH be measured?**

Ans. By a calibrated pH meter using the prescribed soil suspension method.

COMMON VIVA REVISION

IMPORTANT TOPICS

- Normality, molarity, equivalent weight and standard solutions.
- Primary and secondary standards.
- End point and equivalence point.
- Complexometric titration and water hardness.
- Viscosity and the effect of temperature.
- Electrochemical cells, EMF and salt bridges.
- pH, calibration and buffer solutions.
- Condensation polymerisation and thermosetting polymers.
- Dissolved oxygen and its importance in water quality.
- Difference between qualitative and quantitative analysis.

FINAL RECORD CHECKLIST

Item	Completed
Aim written clearly	
Theory understood	
Procedure recorded	
Observation table completed	
Calculation shown with units	
Result written clearly	
Precautions learned	
Viva questions prepared	
Faculty signature obtained	