INORGANIC & PHYSICAL CHEMISTRY

PRACTICAL-I

M.Sc. CHEMISTRY

SEMESTER-I, PAPER-V

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M.Sc. CHEMISTRY: INORGANIC & PHYSICAL CHEMISTRY PRACTICAL-I

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FOREWORD

Since its establishment in 1976, Acharya Nagarjuna University has been forging ahead in the path of progress and dynamism, offering a variety of courses and research contributions. I am extremely happy that by gaining 'A+' grade from the NAAC in the year 2024, Acharya Nagarjuna University is offering educational opportunities at the UG, PG levels apart from research degrees to students from over 221 affiliated colleges spread over the two districts of Guntur and Prakasam.

The University has also started the Centre for Distance Education in 2003-04 with the aim of taking higher education to the door step of all the sectors of the society. The centre will be a great help to those who cannot join in colleges, those who cannot afford the exorbitant fees as regular students, and even to housewives desirous of pursuing higher studies. Acharya Nagarjuna University has started offering B.Sc., B.A., B.B.A., and B.Com courses at the Degree level and M.A., M.Com., M.Sc., M.B.A., and L.L.M., courses at the PG level from the academic year 2003-2004 onwards.

To facilitate easier understanding by students studying through the distance mode, these self-instruction materials have been prepared by eminent and experienced teachers. The lessons have been drafted with great care and expertise in the stipulated time by these teachers. Constructive ideas and scholarly suggestions are welcome from students and teachers involved respectively. Such ideas will be incorporated for the greater efficacy of this distance mode of education. For clarification of doubts and feedback, weekly classes and contact classes will be arranged at the UG and PG levels respectively.

It is my aim that students getting higher education through the Centre for Distance Education should improve their qualification, have better employment opportunities and in turn be part of country's progress. It is my fond desire that in the years to come, the Centre for Distance Education will go from strength to strength in the form of new courses and by catering to larger number of people. My congratulations to all the Directors, Academic Coordinators, Editors and Lesson-writers of the Centre who have helped in these endeavors.

Prof. K. Gangadhara Rao
M.Tech., Ph.D.,
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Acharya Nagarjuna University.

M.Sc. CHEMISTRY
SEMESTER-I, PAPER-V
105CH24 - INORGANIC & PHYSICAL CHEMISTRY
PRATICAL-I

SYLLABUS

LIST OF EXPERIMETNS:

SECTION-I: INORGANIC CHEMISTRY

1) Determination of Zn^{2+} with potassium ferrocyanide (Volumetric).
2) Complexomatric titrations: Determination of Mg^{2+}, Ni^{2+} and hardness of water using EDTA.
3) Determination of Fe^{3+} by photochemical reduction.
4) Argentometry: Determination of chloride by argent metric titration using.
   a) K_2CrO_4 (b) Fluorescein as indicators.

SECTION-II: PHYSICAL CHEMISTRY

1) Relative strengths of acids by studying the hydrolysis of ethylacetate / methyl acetate.
2) Determination of equilibrium constant of KI_3 x $KI + I_2$ by partition coefficient method and determination of unknown concentration of potassium iodide.
3) Distribution coefficient of Benzoic acid between Benzene and water.
4) Determination of critical solution temperature of phenol-water system Study of the effect of eletrolyteon the miscibility of phenol-water system.

Reference Books:

1) Vogels Text Books of Quantitative Analysis, Revised. J. Asset, R.C. Denny, G.H. Jeffery and J. Mendhan. ELBS.
2) Synthesis and Characterisation of Inorganic Compounds, W.L. Jolly. Prentice Hall.
3) Practical Inorganic Chemistry by G. Pass and H. Sutcliffe Chapman and Hall.
4) Practicle Inorganic Chemistry by. K. Somasekhar Rao and K.N.K. Vani.

ACHARYA NAGARJUNA UNIVERSITY: CENTRE FOR DISTANCE EDUCATION

M.Sc. – Chemistry - Program code: 04

Program Structure

Program code	Program	Internal assessment	External exams	Max. Marks	credits
SEMISTER 1					
101CH24	Inorganic Chemistry-I	30	70	100	4
102CH24	Organic Chemistry-I	30	70	100	4
103CH24	Foundation for Chemistry	30	70	100	4
104CH24	Physical Chemistry-I	30	70	100	4
105CH24	Inorganic & Physical Chemistry **Practical-I**	30	70	100	4
106CH 24	Organic Chemistry **Practical-II**	30	70	100	4
SEMISTER 2					
201CH24	Physical Chemistry-II	30	70	100	4
202CH24	Organic Chemistry-II	30	70	100	4
203CH24	Essential Lab Techniques for Industry	30	70	100	4
204CH24	Inorganic Chemistry-II	30	70	100	4
205CH24	Inorganic & Physical Chemistry **Practical-I**	30	70	100	4
206CH24	Organic Chemistry **Practical-II**	30	70	100	4
SEMISTER 3					
301CH24	Applied Inorganic Analysis	30	70	100	4
302CH24	Analysis of Applied Industrial Products	30	70	100	4
303CH24	Optical Thermal & Radiochemical Methods of Analysis	30	70	100	4
304CH24	Principles and Techniques in Classical Analysis	30	70	100	4
305CH24	Classical Methods of Analysis **Practical-I**	30	70	100	4
306CH24	Instrumental Methods of Analysis **Practical-II**	30	70	100	4
SEMISTER 4					
401CH24	Advanced Methods of Analysis	30	70	100	4
402CH24	Analysis of Drugs, Foods, Diary Products & Biochemical Analysis	30	70	100	4
403CH24	Separation Techniques & Electro Analytical Techniques	30	70	100	4
404CH24	Environmental Chemistry & Analysis	30	70	100	4
405CH24	Classical & Instrumental Methods of Analysis **Practical-I**	30	70	100	4
406CH24	Spectral Problems **Practical-II**	30	70	100	4

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M.Sc. DEGREE EXAMINATION, MODEL QUESTION PAPER

M.Sc. CHEMISTRY - FIRST SEMESTER

INORGANIC & PHYSICAL CHEMISTRY (105CH24)

PRACTICAL - I

Max. Marks: 100 (Internal-30M & External-70M)

A total Six (6) Experiments must be carryout taking minimum of ‘3’ in each section

SECTION-I: INORGANIC CHEMISTRY

1) Determination of Zn2+ with potassium ferrocyanide (Volumetric).
2) Complexomatric titrations: Determination of Mg2+, Ni2+ and hardness of water using EDTA.
3) Determination of Fe3+ by photochemical reduction.
4) Argentometry: Determination of chloride by argent metric titration using.
   a) K2CrO4 (b) Fluorescein as indicators.

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1) Relative strengths of acids by studying the hydrolysis of ethylacetate / methyl acetate.
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SECTION-I: INORGANIC CHEMISTRY

EXPERIMENT-1

DETERMINATION OF ZN^{2+} WITH POTASSIUM FERROCYANIDE (PRECIPITATION)

Aim: Determination of Zn^{2+} with potassium ferrocyanide

Objective:

To Determine the concentration of zinc ions (Zn^{2+}) in a solution by titrating with potassium ferrocyanide ($K_4[Fe(CN)_6]$) in the presence of a suitable indicator.

Principle:

Zinc ions react with potassium ferrocyanide to form a white precipitate of zinc ferrocyanide:

$$3Zn^{2+} + 2[Fe(CN)_6]^{4-} \longrightarrow Zn_3[Fe(CN)_6]_2$$

The endpoint is usually detected using a spot test on a white porcelain tile with uranium acetate or sometimes potassium ferricyanide to test for excess ferrocyanide.

Reagents Required:

- Standard Zn^{2+} solution (e.g., zinc sulfate or zinc nitrate)
- Potassium ferrocyanide solution (0.05 M or standardized)
- Dilute acetic acid (5–10%)
- Uranium acetate indicator or potassium ferricyanide solution
- Distilled water

Apparatus Required:

- Burette, pipette, conical flask
- White porcelain spot plate
- Beakers, funnel, glass rod
- Volumetric flask

Procedure:

A. Preparation of Solutions:

1) Zinc standard solution: Dissolve a known amount of pure zinc salt in distilled water to prepare 0.05 M Zn^{2+} solution.

2) Potassium ferrocyanide solution: Prepare a 0.05 M solution by dissolving $K_4[Fe(CN)_6]\cdot 3H_2O$ in distilled water. Standardize if necessary.
3) Indicator solution (optional): Prepare fresh uranium acetate solution or use a freshly made potassium ferricyanide test solution for endpoint detection.

B. Titration Procedure:

1) Pipette 25.0 mL of Zn^{2+} solution into a conical flask.
2) Add 10 mL of dilute acetic acid to maintain a slightly acidic medium.
3) Fill the burette with potassium ferrocyanide solution.
4) Place a few drops of indicator solution or spot 2–3 drops of uranium acetate on a white porcelain tile.
5) Start titrating by slowly adding ferrocyanide solution from the burette into the Zn^{2+} solution, swirling continuously.
6) After each few mL addition, use a glass rod to take a drop from the conical flask and spot it onto the indicator on the tile.
7) The endpoint is reached when a brownish or greenish-blue color appears on the indicator spot, indicating excess ferrocyanide (i.e., all Zn^{2+} has precipitated).
8) Record the burette reading.
9) Repeat for concordant readings.

Observation Table:

S. No.	Volume of Mg^{2+} solution (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume of EDTA Used (mL)
1	25.0			
2	25.0			

Calculations:

From the Reaction Stoichiometry:

$$3Zn^{2+} + 2[Fe(CN)_6]^{4-} \longrightarrow Zn_3[Fe(CN)_6]_2$$

1 mole of potassium ferrocyanide reacts with 1.5 moles of Zn^{2+}.

Use the Titration Formula:

$N_1V_1 = N_2V_2$

Where:

- N_1 = Normality of Zn^{2+}
- V_1 = Volume of Zn^{2+} solution (25 mL)
- N_2 = Normality of potassium ferrocyanide
- V_2 = Volume of ferrocyanide used (from burette)

If molarities are used, account for the reaction stoichiometry accordingly.

Then calculate concentration of Zn^{2+} and convert to mg/L if needed using molar mass of Zn (65.38 g/mol)

Result:

The concentration of Zn^{2+} in the given solution is: __ mol/L or mg/L (based on calculation).

Precautions:

- Potassium ferrocyanide should be freshly prepared and filtered if needed.
- Ensure that all glassware is clean and free of Zn contamination.
- Always test the reaction drop immediately — don't let it dry before reading.
- Do not overshoot the endpoint; watch closely for the first appearance of color on the tile.
- Avoid using strongly acidic or basic solutions as it may decompose ferrocyanide.

EXPERIMENT-2A

DETERMINATION OF MAGNESIUM IONS (MG^{2+}) - COMPLEXOMETRIC TITRATION

Aim: Determination of Magnesium Ions (Mg^{2+}) by Using EDTA

Principle:

EDTA (ethylenediaminetetraacetic acid) forms a stable 1:1 complex with Mg^{2+} ions:

$$Mg^{2+} + EDTA^{4-} \longrightarrow [MgEDTA]^{2-}$$

The endpoint is detected using Eriochrome Black T (EBT) indicator in an ammonia buffer (pH ~10). At this pH, EBT forms a weak wine-red complex with Mg^{2+}, which turns blue once Mg^{2+} is completely complexed by EDTA.

Reagents Required:

1) Standard EDTA solution (0.01 M)
2) Magnesium sulfate ($MgSO_4$) solution (unknown concentration)
3) Ammonia-ammonium chloride buffer (pH 10)
4) Eriochrome Black T indicator (EBT)
5) Distilled water

Apparatus:

- Burette
- Pipette (25 mL)
- Conical flask (250 mL)
- Volumetric flask
- Beaker

Procedure:

1) Pipette 25.0 mL of the Mg^{2+} solution into a clean conical flask.
2) Add 5 mL of ammonia buffer (pH 10) to maintain basic conditions.
3) Add 2–3 drops of Eriochrome Black T indicator:
    - The solution will turn wine red due to the Mg–EBT complex.

4) Titrate with 0.01 M EDTA solution from the burette:
    - Swirl continuously.
    - Endpoint: color changes from wine red to pure blue.
5) Note the volume of EDTA used.
6) Repeat for concordant readings (±0.1 mL difference).

Calculation:

EDTA and Mg^{2+} react in a 1:1 molar ratio:

Moles of EDTA = M_{EDTA} X V_{EDTA}

Moles of Mg^{2+} = Moles of EDTA

Then:

$$\text{Concentration of } Mg^{2+} = \frac{\text{Moles of } Mg^{2+}}{\text{Volume of sample (L)}}$$

Observation Table:

S. No.	Volume of Mg^{2+} solution (mL)	Initial Burette Reading (mL)	Final Burette Reading (mL)	Volume of EDTA Used (mL)
1	25.0			
2	25.0			

Result:

The concentration of Mg^{2+} in the given solution is: __ mol/L or mg/L (based on calculation).

EXPERIMENT-2B

DETERMINATION OF NICKEL ION (NI^{2+}) - COMPLEXOMETRIC TITRATION

Aim: Determination of Nickel (Ni^{2+}) Using EDTA

Principle:

EDTA (ethylenediaminetetraacetic acid) forms a 1:1 stable complex with Ni^{2+}:

Ni^{2+} + $EDTA^{4}$- $\longrightarrow$ $[Ni–EDTA]^{2-}$

At alkaline pH (~10), the reaction proceeds completely. An indicator such as Murexide is used, which changes color from yellow to violet at the endpoint.

Reagents Required:

1) 0.01 M EDTA solution (standard)
2) $NiSO_4$ solution (unknown Ni^{2+} concentration)
3) Ammonia-ammonium chloride buffer (pH 10)
4) Murexide indicator (purple color in free state)
5) Distilled water

Apparatus:

- Burette
- Pipette (25 mL)
- Conical flask
- Volumetric flask
- Beaker

Procedure:

1) Pipette 25.0 mL of the nickel solution into a conical flask.
2) Add 5 mL of pH 10 buffer solution to maintain alkaline pH.
3) Add a small pinch of Murexide indicator:
    - The solution turns yellow in presence of Ni^{2+}.

4) Titrate with 0.01 M EDTA from the burette:
   - Swirl continuously
   - Endpoint: Color changes from yellow to violet.
5) Note the volume of EDTA used.
6) Repeat for concordant readings.

Observation Table:

S. No.	**Volume of Ni^{2+} solution (mL)**	**Initial Burette Reading (mL)**	**Final Burette Reading (mL)**	**Volume of EDTA Used (mL)**
1	25.0			
2	25.0			

Calculation:

Since the reaction is 1:1:

Moles of EDTA = M_{EDTA} X V_{EDTA}

Moles of Ni^{2+} = Moles of EDTA

Then:

$$\text{Concentration of } Ni^{2+} = \frac{\text{Moles of } Ni^{2+}}{\text{Volume of sample (L)}}$$

You may convert it into mg/L using molar mass of Ni (58.69 g/mol).

Result:

The concentration of Ni^{2+} in the given solution is: _____ mol/L or mg/L (as per calculation).

EXPERIMENT-2C

DETERMINATION OF TOTAL HARDNESS OF WATER

Aim: To determine the total hardness of the given water sample by complexometry.

Apparatus: Burette-50ml, conical flask-250ml, Measuring cylinder (5ml)

Chemicals Required:

Disodium salt of EDTA, ammonium chloride, ammonium hydroxide, eriochrome black–T (EBT).

Theory:

The hardness of the water is generally due to the presence of calcium and magnesium ions. Ions like Ca2+ and Mg2+ have a tendency to form complexes with EDTA. Certain dyestuffs which form weak coloured complexes with cations can serve as visual complexometric indicators. For titrimetric determination of such metal ions, first the metal - indicator complex is formed which is wine red in colour.

M + + In ⟶ M–In complex

(Metal) (Indicator) Wine red

During titrations with EDTA, the metal ions are progressively complexed by EDTA. At final the indicator is displaced from metal-indicator (M-In) complex leaving free indicator. The colour change accompanying this progress gives the end point.

M–In complex + EDTA ⟶ M–EDTA complex + In

Wine red Blue

Preparation of Reagents:

Preparation of Standard EDTA Solution:

Disodium salt of ethylene diamine tetra acetic acid is available commercially and can be used as primary standard. The commercial analytical reagent may contain a trace of moisture. So it is dried at 800C. Then its composition agree exactly with the formula Na2H2C10H12.2H2O with molecular weight 372.5. About 0.9314 g of disodium salt of EDTA is weighted accurately and dissolved in 250 ml of distilled water in a volumetric flask.

Preparation of buffer solution (pH=10):

7 gm of ammonium chloride is dissolved in 56.8 ml of liquor ammonia (i.e., ammonium hydroxide) and diluted to 100 ml.

Preparation of eriochromeblack-T (EBT):

0.2 gm of eriochromeblack–T dyestuff is dissolved in 15 ml of triethanolamine and 5 ml of absolute alcohol is added to reduce viscosity. The indicator is stable for several months.

(or)

4.0 gm of eriochromeblack–T dyestuff is dissolved in 100 ml of methanol, which is stable for atleast one month.

Procedure:

By the aid of a burette, a 50 ml of the given water sample is transferred in to a 250 ml conical flask. About 4 ml of buffer solution and two drops of EBT indicator are added. The wine red solution is titrated against standard EDTA solution taken in a burette. The end point is wine red to blue.

The experiment is repeated till two concurrent values are obtained and the total hardness is calculated in terms of calcium carbonate.

OBSERVATIONS AND CALCULATIONS:

Weight of Disodium ethylene diamine tetra acetate (w) = ________ g in 250 ml

Molarity of Disodium ethylene diamine tetra acetate solution (M_1)

$$= \frac{\text{Wt. of EDTA salt X 1000}}{\text{Eq. wt. of EDTA salt X Volume}}$$

$\underline{\text{W X 1000}}$ = ________ M

$$= \text{372.25X250}$$

Total Hardness Determination:

S.No.	Vol. of hard water transferred (V_2 ml)	Burette Reading		Volume of standard EDTA sol. Rundown (V_1 ml)
		Initial	Final	
1	50.0			
2	50.0			
3	50.0			

We know that $V_1M_1 = V_2M_2$

Where , V_1 = Volume of standard EDTA solution = ______ M

M_1 = Molarity of EDTA solution = ______ M

V_2 = Volume of hard water = 50 ml

M_2 = Molarity of hard water (in terms of $CaCO_3$)= ?

Therefore, $M_2 = \frac{V_1M_1}{V_2}$ = __________M

Total hardness of water (in terms of $CaCO_3$)

= Molarity of water sample w.r.t. $CaCO_3$ X Mol.Wt. of $CaCO_3$ X 1000 mg/lit

= M2 X 100.09 X 1000 = _______ mg/lit

Total hardness of water (in terms of $CaCO_3$) = mg / lit

Method of Calculation:

Total hardness of water is determined in terms of amount of $CaCO_3$ present in million parts of water. Calcium and magnesium forms a 1:1 complex with EDTA.

Result: The total hardness of the given water sample is __________ ppm.

Precautions:

1) The titration should be carried out slowly with the shaking after addition of each drop of EDTA solution, because of the slow formation of complexes.
2) Buffer solution should be freshly prepared.
3) Addition of excess indicator should be avoided.

EXPERIMENT-3

DETERMINATION OF FE^{3+} BY PHOTOCHEMICAL REDUCTION

Aim: Determination of Fe^{3+} by Photochemical Reduction Using Potassium Dichromate as Oxidant

Principle:

Fe^{3+} ions are photochemically reduced to Fe^{2+} upon exposure to light in acidic medium. The Fe^{2+} formed is then estimated volumetrically by titrating against a standard potassium dichromate ($K_2Cr_2O_7$) solution using diphenylamine sulfonate as an internal indicator.

$$Fe^{3+} + \longrightarrow Fe^{2+}$$

The Fe^{2+} reacts with dichromate as:

$$6Fe^{2+} + Cr_2O^{2-}{}_7 + 14H^+ \longrightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$$

Apparatus:

- Volumetric flasks
- Conical flasks
- Burette
- Pipette
- Light source (sunlight or UV lamp)
- Beakers

Reagents Required:

1) $FeCl_3$ solution (unknown concentration)
2) 0.01 N potassium dichromate ($K_2Cr_2O_7$) solution
3) Dilute sulfuric acid (H_2SO_4)
4) Diphenylamine sulfonate indicator
5) Distilled water

Procedure:

1) Take 50 mL of $FeCl_3$ solution in a beaker.

2) Add 10 mL of dilute H_2SO_4 to acidify the solution.
3) Place the beaker in sunlight or under a UV lamp for 30–60 minutes to allow photochemical reduction of Fe^{3+} to Fe^{2+}.
4) After irradiation, transfer the solution into a conical flask.
5) Add 3–4 drops of diphenylamine sulfonate indicator - solution appears violet-blue.
6) Titrate with 0.01 N $K_2Cr_2O_7$ from a burette:
   - Swirl the solution during titration.
   - Endpoint: color changes from violet-blue to light green (disappearance of blue).
7) Note the volume of $K_2Cr_2O_7$ used.
8) Repeat the titration for concordant readings.

Observation Table:

S. No.	**Volume of Fe^{3+} solution (mL) V_2**	**Initial Burette Reading (mL)**	**Final Burette Reading (mL)**	**Volume of Dichromate Used (mL) V_1**
1	50.0			
2	50.0			

Calculation:

From the redox reaction:

$$6Fe^{2+} + Cr_2O_7^{2-} \longrightarrow 6Fe^{3+} + 2Cr^{3+}$$

So, 1 mole of $Cr_2O_7^{2-}$ ≡ 6 moles of Fe^{2+}

Use:

$$N_1V_1 = N_2V_2$$

Where:

- N_1, V_1 = Normality and volume of dichromate
- N_2, V_2 = Normality and volume of Fe^{2+}

Result:

The concentration of Fe^{3+} in the given solution is calculated as: _____ mol/L or mg/L

EXPERIMENT-4A

ESTIMATION OF CHLORIDE IN WATER

Aim: To determine the chloride content of the given sample of water using silver nitrate solution with potassium chromate as an indicator (Mohr's indicator).

Apparatus required: Pipette, Burette, Conical flask, Burette Stand, Wash bottle, Glazed tile, volumetric flask, beaker and dropper.

Chemicals required:

a) Standard sodium chloride solution.

b) Silver nitrate solution.

c) Potassium chromate solution.

d) Water sample.

Chemical Reactions:

$AgNO_3 + Cl \longrightarrow - AgCl + NO_3^-$

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

Yellow Red

Procedure:

Part A: Standardization of Silver nitrate solution:

10 ml of the standard sodium chloride is pipetted into a conical flask keeping it upon a tile. To it 1 ml of the indicator solution is added. The solution colour changes to yellow. This solution is titrated slowly with silver nitrate solution from the burette, swirling the solution constantly until the red color formed by the addition of each drop begins to disappear more slowly. This is an indication that more of the chloride has been precipitated, change in the color occurs. At the end point a faint reddish tinge persists after brisk shaking. The final burette reading is noted. The titration is repeated until a concordant value is obtained. The molarity of silver nitrate solution is calculated.

Part B: Estimation of Chloride content in water:

20 ml of the water sample is pipetted into a conical flask keeping it upon a tile. To it 1ml of the indicator solution is added. The solution colour changes to yellow. This solution is titrated

slowly with silver nitrate solution from the burette, swirling the solution constantly until the red color formed by the addition of each drop begins to disappear more slowly. This is an indication that more of the chloride has been precipitated, change in the color occurs. At the end point a faint reddish tinge persists after brisk shaking. The final burette reading is noted. The titration is repeated until a concordant value is obtained.

Observations & Calculations:

Part A: Standardization of Silver Nitrate:

Molarity of NaCl =M1=

Sl. No.	**Volume of Sodium Chloride V_1 ml**	**Burette readings**		**Volume of Silver Nitrate solution (b-a) V_2 ml**
		Initial (a)	**Final (b)**	
1.	10			
2.	10			
3.	10			

Volume of Sodium chloride = V_1 ml

Molarity of Sodium chloride = M_1

Volume of Silver nitrate = V_2 ml

Molarity of Silver nitrate = M_2

n_1= No. of moles of NaCl in the balanced equation = 1

n_2 =No. of moles of $AgNO_3$ in the balanced equation = 1

$V_1M_1/n_1 = V_2M_2/n_2$

$$M2 = \frac{V_1M_1 \text{ x } n_2}{n_1V_2}$$

Therefore molarity of silver nitrate solution (M_2) =

Part B: Estimation of Chloride in water:

Morality of silver nitrate solution = M2=

Sl. No.	**Volume of water sample (in ml)**	**Burette Readings**		**Volume of Silver Nitrate used (b-a) V_3 ml**
		Initial (a)	**Final (b)**	
1.	20			
2.	20			
3.	20			

1000 ml of 1M $AgNO_3$ = 35.46 grams of chloride

V3 ml of M_2 $AgNO_3$ is equivalent to $\frac{V_3M_2 \times 35.46}{1000}$ grams of chloride

20 ml of water sample contains $\frac{V_3M_2 \times 35.46}{1000}$ grams = X grams of chloride

1000 ml of water sample contains 50 x X grams of chloride

5×10^4 x X mg of Cl-

Chloride content of given water sample = ppm

Result: Chloride content of given water sample… .... ppm

EXPERIMENT-4B

DETERMINATION OF CHLORIDE ION BY ARGENTOMETRIC TITRATION

Aim: Determination of Chloride Ion by Argentometric Titration Using Fluorescein Indicator (Fajans Method)

Principle:

Chloride ions react with silver nitrate ($AgNO_3$) to form a white precipitate of AgCl:

$$Ag^+ + Cl^- \longrightarrow AgCl\,(s)$$

When all chloride has reacted, excess Ag^+ ions adsorb onto the surface of AgCl particles. In presence of fluorescein, a negatively charged dye, this leads to the formation of a pink-colored complex on the surface of the precipitate — marking the endpoint.

Type of Titration:

Adsorption indicator titration (Fajans method)

Reagents Required:

1) Standard $AgNO_3$ solution (0.01 N)
2) Chloride solution (NaCl or unknown sample)
3) Fluorescein indicator (0.1% aqueous or alcoholic solution)
4) Nitric acid (HNO_3) – dilute
5) Distilled water

Apparatus:

- Burette
- Pipette (25 mL)
- Conical flask (250 mL)
- Beakers
- Glass rod

Procedure:

1) Pipette 25.0 mL of the chloride-containing solution into a conical flask.
2) Add 1–2 mL of dilute nitric acid to acidify the solution (prevents interference from

carbonate ions).

3) Add 2–3 drops of fluorescein indicator - the solution will appear pale yellow-green.
4) Titrate with standard $AgNO_3$ solution from the burette:
    - Swirl continuously.
    - A pale pink color appears at the endpoint.
5) Record the volume of $AgNO_3$ used.
6) Repeat to get concordant readings.

Observation Table:

S. No.	**Volume of Cl^- solution (mL) V_2**	**Initial Burette Reading (mL)**	**Final Burette Reading (mL)**	**Volume of $AgNO_3$ Used (mL) V_1**
1	25.0			
2	25.0			

Reaction:

$$AgNO_3 + NaCl \longrightarrow AgCl\,(s) + NaNO_3$$

Calculation:

Using:

$$N_1V_1 = N_2V_2$$

Where:

- N_1, V_1= Normality and volume of $AgNO_3$
- $N_2\,V_2$= Normality and volume of Cl^- solution

Then find chloride concentration in mol/L or mg/L.

Result:

The concentration of chloride ions (Cl^-) in the sample is: _____ mol/L or mg/L

SECTION-II:

PHYSICAL CHEMISTRY

SEMESTER- I

1) Relative strengths of acids by studying the hydrolysis of ethylacetate / methyl acetate.
2) Determination of equilibrium constant of $KI_3 \rightarrow KI + I_2$ by partition coefficient method and determination of unknown concentration of potassium iodide.
3) Distribution coefficient of Benzoic acid between Benzene and water.
4) Determination of critical solution temperature of phenol-water system Study of the effect of electrolyte on the miscibility of phenol-water system.

REFERENCE BOOKS:

1) Vogels Text Books of Quantitative Analysis, Revised. J. Asset, R.C. Denny, G.H. Jeffery and J. Mendhan. ELBS.

EXPERIMENT-1

RELATIVE STRENGTHS OF ACIDS BY STUDYING THE HYDROLYSIS OF ETHYLACETATE / METHYL ACETATE. [ESTER HYDROLYSIS]

Aim:

To determine the relative strength of two acids by ester hydrolysis

Apparatus:

1) Conical flask.
2) Burette.
3) Pipette.

Chemicals Required:

1) Methyl Acetate
2) Hydrochloric Acid
3) Sodium Hydroxide (NaOH)
4) Oxalic Acid.
5) Phenolphthalein.

Preparation of Solutions:

a) 1N and 2N HCl: 22.5 mL and 45 mL of Conc. HCl is taken in a 250 mL volumetric flask and make up them up to the mark with distilled water.
b) 0.5N NaOH solution: 10 g of NaOH is taken in the 500 mL volumetric flask. Dissolve it in distilled water and make up to the mark with distilled water.
c) 0.5N Oxalic Acid: Accurately 6.3 g of oxalic acid is weighed and transferred into 100 mL volumetric flask. The substance is dissolved in distilled water and make up to the mark with distilled water.

Principle:

The hydrolysis of Methyl acetate or Ethyl acetate in presence of an acid may be represented as

$$CH_3COOCH_3 + H_2O \xrightarrow{H^+} CH_3COOH + CH_3OH$$

Methyl acetate Acid Alcohol

$$CH_3COOC_2H_5 + H_2O \xrightarrow{H^+} CH_3COOH + C_2H_5OH$$

Ethyl acetate Acid Alcohol

The Rate of reaction is given by

$$\left(\frac{Dx}{Dt}\right) = \left(\frac{2.303}{t}\right) \times \log\left(\frac{a}{(a-x)}\right) \rightarrow (1\ order)$$

As Acetic acid is produced during the reaction, the reaction may be studied by the titration of unknown concentration of reaction mixture with a standard Alkali at suitable intervals of time.

The increasing in the acidity of the solution will have increase of amount of alcohol produced and hence the amount of methyl acetate or ethyl acetate is hydrolysed.

$V_\infty = V_0$ is directly proportional to 'a'

$(V_\infty = V_t)$ is directly proportional to (a-x)

$$X = \left(\frac{2.303}{t}\right) \times \log\left(\frac{V_\infty - V_0}{V_\infty - V_t}\right)$$

Procedure:

Standardize 1N and 2N HCl by Known Concentration of NaOH solution, 100 mL of 1N HCl is taken in a conical flask and 10 mL of pure Methyl acetate/ Ethyl acetate is taken in a test tube. Both the Conical flask and the test tubes are placed in water bath to bring them into room temperature. Measure exactly 5mL of Methyl acetate/ Ethyl acetate and add to 100 mL of 1N HCl in a Conical flask with Stopper.

When exactly half of the amount in the pipette is been discharged, the stopwatch is started. Mixture is shaken well and maintain at a constant Temperature. Now titrated with NaOH solution using phenolphthalein as indicator. The volume of Alkali required for titration is noted and is taken as "V_o". Similarly, 5 mL of reaction mixture is taken out in successive intervals of time 10, 20, 30, 40 and 50 minutes. 5ml of reaction mixture is taken in a conical flask and 20 mL of ice -cold water and titrated with NaOH using Phenolphthalein as indicator.

To obtain complete hydrolysis, the reaction mixture is kept in a water bath maintained at 50 °C for about half an hour. After the hydrolysis is completed, it is cooled at room temperature and titrated the 5 mL of the reaction mixture with NaOH Solution and is taken as, V∞.

Method of Calculation:

The initial volume of titrant, NaOH is V_0 at T_0 ^oC is proportional to the amount of HCl present in the reaction mixture at zero time.

When there is an Acetic Acid present as the hydrolysis, proceeds V`-Vo, directly proportional to initial concentration of ester. At successive intervals of time $V`-V_t$ is proportional to the concentration of esters at time "t".

$$K = (\frac{2.303}{t})\ X\ log\ \frac{(V - V0)}{(V\infty - Vt)} sec - 1$$

Same procedure and calculation are repeated for 2N HCl solution

Relative Strength of two acids is given by K_1/K_2

Report:

The Rate Constant for Hydrolysis of Methyl Acetate/Ethyl acetate by 1N HCl and 2N HCl are

Rate constant of 1N HCl, K_1 = _______sec $^{-1}$

Rate constant of 2N HCl, K_2 = _______sec $^{-1}$

Relative strength of acids = K_2/K_1 =

Calculations:

1. **Normality of Oxalic acid :**

$$N_1 = \frac{\text{Amount of Oxalic acid}}{\text{Gram Equivalent weight of Oxalic acid (63.035)}} \text{ X } \frac{1000}{250}$$

= -----------------------N

2. Standardization of Sodium Hydroxide Solution:

S.No.	Volume of Oxalic acid (V_1) mL	Burette Readings		Volume NaOH solution (V_2) mL
		Initial	Final	
1	20 mL			
2	20 mL			
3	20 mL			

Where,

Volume of Oxalic acid Solution V_1 = 20.0 mL

Normality of Oxalic acid Solution N_1 = _____N

Volume of Sodium Hydroxide solution V_2 = _____mL

Normality of Sodium Hydroxide solution N_2 = _____N

We know that $V_1N_1 = V_2N_2$

Therefore,

$$N_2 = \frac{N_1V_1}{V_2}$$

3. Calculations for determining Rate constants:

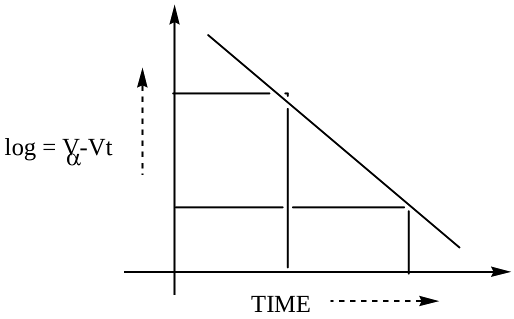
For 1 N HCl :

S.No.	Time(t) Minutes	Burette initial reading (ml)	Burette final reading (ml)	Volume of NaOH Consume (V_t ml)	$(V_\infty - V_t)$ml	$\text{Log}(V_\infty - V_t)$	$K_1 = (2.303/t) \times \text{Log}(V_\infty - V_0)/(V_\infty - V_t)$
1	0						
2	10						
3	20						
4	30						
5	40						
6	50						
7	60						
8	V_∞						

Average K_1 =

V_0 = ____________mL, V_∞ = ______mL

$V_0 - V_\infty$ = ______mL



From Graph Slope = _______________

$$\text{Slope} = \frac{K_1}{2.303} \Rightarrow K_1 = \text{Slope} \times 2.303$$

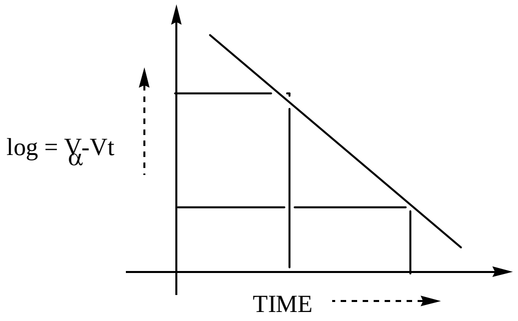
For 2N HCl :

S.No.	Time(t) Minutes	Burette initial reading (ml)	Burette final reading (ml)	Volume of NaOH Consume (V_t ml)	$(V_\infty - V_t)$ml	$\text{Log}(V_\infty - V_t)$	$K_2 = (2.303/t) \times \text{Log}(V_\infty - V_0)/(V_\infty - V_t)$
1	0						
2	10						
3	20						
4	30						
5	40						
6	50						
7	60						
8	V_∞						

Average K_2 =

V_0 = ____________mL, V_∞ = ______mL

$V_0 - V_\infty$ = ______mL



From Graph Slope = _______________

$$\text{Slope} = \frac{K_1}{2.303} \Rightarrow K_1 = \text{Slope} \times 2.303$$

Report:

Rate constant of 1N HCl, K_1 = _______sec $^{-1}$

Rate constant of 2N HCl, K_2 = _______sec $^{-1}$

Relative strength of acids = K_2/K_1 =

EXPERIMENT-2

DETERMINATION OF EQUILIBRIUM CONSTANT OF $KI_3 \rightarrow KI + I_2$ BY PARTITION COEFFICIENT METHOD AND DETERMINATION OF UNKNOWN CONCENTRATION OF POTASSIUM IODIDE

Aim: To determine the equilibrium constant for the equilibrium. $KI_3 \rightarrow KI + I_2$

Chemicals required:

1) 1% Iodine in CCl_4
2) Carbon Tetra Chloride
3) 0.05 N of KI in 250 mL
4) 0.01 N Hypo Solution
5) Starch Indicator in 100 mL
6) 10% KI in 100 mL
7) 0.01 N Potassium Dichromate solution in 250 mL

Preparation of Solutions:

0.05 N KI solution in 250 mL: About 2.125gms of Potassium Iodide is weighed accurately and then transferred in to 250ml standard flask through funnel. The substance is dissolved in minimum quantity of water and then made up to the mark by adding distilled water.

1% Iodine in CCl_4: 1g Iodine is weighed accurately and dissolved in 100 mL CCl_4.

0.1N Hypo: About 24.819 gm of sodium thiosulphate pentahydrate is weighed accurately.

And then transferred into a 500 mL standard flask through funnel. The substance is dissolved in minimum quantity of water and then made up to the mark by adding distilled water

Starch Indicator: About 1 g of starch powder is weighed and present with few mL of cold water and pour this solution drop by drop into 100 mL of boiling water and boil for few minutes.

10% Potassium Iodide: 10 g of potassium Iodide is weighed and dissolved into 100 mL distilled water.

0.1N Potassium dichromate:

About 1.114 g of pure potassium dichromate is weighed accurately and then transferred into a 250 mL conical flask through funnel wash the funnel with water dissolve the substance and made up to the mark.

Principle: When KI solution is added to the solution of I_2, they react to form KI_3. The Equilibrium state exists between I_2 and KI to form KI_3.

$$I_2 + KI \rightleftharpoons KI_3$$

Equilibrium constant

$$K = \frac{[KI_3]_{Eq}}{[KI]_{Eq} \times [I_2]_{Eq}}$$

On determining $[KI_3]_{(Eq)}$, $[KI]_{(Eq)}$, $[I_2]_{(Eq)}$ we can calculate Equilibrium constant.

The concentration of iodine $[I_2]_{(Eq)}$ can be determined by the partition co-efficient method.

Procedure:

Standardisation of Sodium Thiosulphate: 20 mL of potassium dichromate solution is pipetted out into a conical flask and add 5 mL of 4N HCl and 10 mL of 10% KI are added, now the conical flask is covered with watch glass and kept kin dark room for few minutes the liberated iodine dissolves in the excess of potassium iodide present in it. Then the sol is diluted to about 100 mL of distilled water. Now titrate the solution with Hypo till the brown colour of the solution changes to straw yellow colour. Then one or two ml of starch solution is added then the solution turns to blue coloured again titrate this solution against sodium thiosulphate solution until the blue colour changes to green colour. This is the end point. Repeat the titration until the concurrent values are obtained.

Determination of Equilibrium constant for

$$I_2 + KI \rightleftharpoons KI_3$$

Take two 250 mL Iodine flasks marked as A and B. Take 30 mL, 40 mL of Iodine (I_2) in CCl_4 in flasks A and B respectively. Make the volumes of the flasks up to 50 mL with CCl_4. Add 50 mL of 0.05N KI solution to each flask. Fix stopper to the flask and shake about 90 to 120 minutes. A separating funnel separates Organic layer aqueous layers. The concentration of I_2 in each phase is calculated by titrating with 0.01N hypo solution.

Determination of Concentration of I_2 in Organic Layer: Pipette out 5 mL of Organic layer. Add 5 mL of 10% KI solution. Shaken for few minutes and add 10 mL of water. Titrating with 0.01N hypo solution till the solution turns to straw yellow in colour then add one to two ml of starch indicator and continue the titration until the deep blue coloured solution changes to colourless solution.

Determination of Concentration of I_2 in Aqueous Layer : Pipette out 10 mL of aqueous layer and add 10 mL of water and titrating with 0.01N hypo solution till the solution turns to straw yellow in colour then add one to two mL of starch indicator and continue the titration until the deep blue coloured solution changes to colourless solution.

Then the rate of Equilibrium or Equilibrium constant

$$K = \frac{[KI_3]_{Eq}}{[KI]_{Eq}, [I_2]_{Eq}}$$

Report:

The Equilibrium constant of the reaction is ____

Calculations:

Weight of weighing bottle substance W_2 = __________g

Weight of bottle after transferring the substance W_1 = _______g

Weight of the substance transferred $W = W_2-W_1$

$$\text{The concentration of potassium dichromate} = \frac{(W_2-W_1) \times 0.1}{1.225}$$

Standardisation of Sodium Thiosulphate:

S.No.	**Volume of Potassium dichromate (mL)**	**Burette Readings**		**Volume of hypo consumed (V_2) mL (0.1N)**
		Initial (mL)	**Final (mL)**	
1	20 mL	0 mL		
2	20 mL	0 mL		
3	20 mL	0 mL		

Volume of Potassium dichromate V_1 = 20 mL

Concentration of Potassium dichromate N_1 = N

Volume of Hypo V_2 = mL

Concentration of Hypo N_2 =

$$V_1N_1 = V_2N_2$$

$$\mathbf{N_2 = \frac{N_1V_1}{V_2}}$$

Concentration of Hypo =

Titration of Organic Layer:

S.No.	Volume of Organic layer (mL)	Burette Readings		Volume of hypo consumed (V_2) mL (0.1 N)
		Initial (mL)	Final (mL)	
1	5 mL	0 mL		
2	5 mL	0 mL		
3	5 mL	0 mL		

Volume of Hypo V_2 = mL

Concentration of Hypo N_2 = 0.1 N

Volume of Organic layer V_3 = 5 mL

Concentration of Organic layer N_3 =

$$V_2N_2 = V_3N_3$$

$$\mathbf{N_3 = \frac{N_2V_2}{V_3}}$$

Concentration of $[I_2]_{org}$ =

$$\text{Partition Co-efficient} = \frac{[I_2]_{Org}}{[I_2]_{Eq}}$$

$$[I_2]_{Eq.\ free} = \frac{[I_2]_{Org}}{\text{Partition Corfficient}}$$

Titration of Aqueous Layer:

S.No.	Volume of Aqueous layer (mL)	Burette Readings		Volume of hypo consumed (V_2) mL (0.1) N
		Initial (mL)	Final (mL)	
1	10 mL	0 mL		
2	10 mL	0 mL		
3	10 mL	0 mL		

$$[I_2]_{org} \leftrightarrow [I_2]_{aq}$$

Volume of Hypo V_2 = mL

Concentration of Hypo N_2 =

Volume of Iodine in Aqueous layer V_4 = mL

Concentration of Iodine in Aqueous layer N_4 =

$$V_2N_2 = V_4N_4$$

$$\mathbf{N_4 = \frac{N_2V_2}{V_4}}$$

$$\text{Concentration of } [I_2]_{Aq\ Total} = \frac{\text{(Con. of hypo) x (Volume of hypo)}}{\text{Volume of Organic layer) x (2)}}$$

Concentration of KI [KI] = _____ N

Concentration of Hypo solution = ____ N

Partition Co-efficient = 85.5

$$[I_2]_{\text{Eq free}} = \underline{\hspace{3cm}}\ \text{N}$$

$$[I]_{\text{Aq total}} = [I_2]_{\text{Eq total}}$$

$$[KI_3]_{\text{Eq}} = [I_2]_{\text{Eq total}} - [I_2]_{\text{Eq Free}}$$

$$[KI_3]_{\text{Eq}} = [KI] - [KI_3]_{\text{Eq}}$$

$$K = \frac{[KI_3]_{\text{Eq}}}{[KI]_{\text{Eq}} \text{ X } [I_2]_{\text{Eq}}}$$

Equilibrium constant,

EXPERIMENT-3

DISTRIBUTION COEFFICIENT OF BENZOIC ACID BETWEEN BENZENE AND WATER

Aim: To determine the partition coefficient of benzoic acid between benzene and water.

Required Chemicals:

1) Saturated solution of benzoic acid in benzene
2) Benzene
3) 0.01N NaOH
4) 0.1N NaOH
5) Distilled water

Apparatus

1) Separating funnel: 250 ml
2) Conical flask
3) Pipette
4) Burette
5) Stoppered bottles

Principle:

When a substance is added to a system containing two immiscible liquids, it distributes between them in a definite ratio." This is called Nernst distribution law. The added substance should have solubility in the two liquids for distribution. This is known as the partition coefficient K of a substance between two liquids, given by the formula.

$$K = \frac{\text{Concentration of substance in organic layer}}{\text{Conentartion of substance in aqueous layer}} = \frac{C_1}{C_2}$$

The distribution of benzoic acid between benzene and water is studied in the present experiment. Benzoic acid is an organic substance and has high solubility in benzene. It has less solubility in water. As a result, benzoic acid will partition preferably into benzene layer. The formula for calculating the benzoic acid partition coefficient between benzene and water is given below. C_1 and C_2 are concentration of benzoic acid in organic and aqueous layer. In the present experiment, benzoic acid is shaken with benzene and water for 30 minutes to achieve distribution. Shaking is required to achieve distribution equilibrium. At equilibrium the speed of forward process is equal to the speed of backward process. Benzoic acid is distributed as associated molecules in benzene layer and un associated molecules in aqueous layer. Hence, the equation is given as follows.

$$\mathbf{K} = \frac{\sqrt{\text{Concentration of substance in organic layer}}}{\text{Concentration of substance in aqueous layer}} = \frac{\sqrt{C1}}{C2}$$

The partition coefficient K will be remains constant only if there is neither association nor dissociation of solute molecules in both the phases.

Procedure:

Preparation of 0.1N Sodium hydroxide: 4 gm of sodium hydroxide was dissolved in 1000 mL of distilled water and make up the final volume in volumetric flask.

1) Weigh the samples (250 mg, 500 mg and 750 mg) of benzoic acid into three reagent bottles and add 50 mL of benzene and 50 mL of water to all three reagent bottles.
2) Keep the bottles on constant temperature water bath and Shake the bottles for 30 minutes.
3) Transfer the contents into a separating funnel and allow them to separate as two layers.
4) Collect the aqueous layer and titrate 10 mL of sample with 0.1N sodium hydroxide solution using phenolphthalein as indicator.
5) Similarly collect the organic layer (benzene) and titrate 10 mL of sample with 0.1N sodium hydroxide solution using phenolphthalein as indicator.
6) Calculate the partition coefficient of benzoic acid between benzene and water.

Observations and Calculations:

Equivalent factor: Each mL of 0.1N sodium hydroxide = 0.0122 gm of benzoic acid

Concentration of benzoic acid = Volume of sodium hydroxide consumed x 0.0122

S.No.	**The volume of aqueous / benzene layer taken**	**The volume of sodium hydroxide consumed in mL**	**Concentration of benzoic acid**	$\sqrt{C}$	**Partition coefficient** $\sqrt{C_1}/C_2$
1	10 mL organic		C_1=		
2	10 mL organic		C_1=		
3	10 mL aqueous		C_2=		
4	10 mL aqueous		C_2=		

Report: The partition coefficient of benzoic acid between benzene and water is………………….

EXPERIMENT-4

DETERMINATION OF CRITICAL SOLUTION TEMPERATURE OF PHENOL - WATER SYSTEM STUDY OF THE EFFECT OF ELECTROLYTE ON THE MISCIBILITY OF PHENOL - WATER SYSTEM

Aim: To determine the effect of electrolyte on Critical Solution Temperature of Phenol–Water System.

Chemicals Required:

1) 80% Phenol = 80 mL Phenol + 20 mL water.

2) 0.1 N NaCl

Procedure:

From 0.1N NaCl solution 0.02N, 0.04N, 0.06N, 0.08N NaCl solutions are prepared. In a boiling tube 5 mL of Phenol solution and 5 mL of 0.02 N NaCl are taken. The boiling tube with stirrer and thermometer is placed in a beaker in which water will be taken and the boiling tube is clamped to retort stand. On stirring the solution, white cloudiness is appeared. On heating the solution gradually at certain temperature, the cloudiness disappears. The temperature is noted. Again, on continues cooling, at certain temperatures a clear solution becomes cloudy. The temperature is noted. The average of these two temperatures will gives the Critical Solution Temperature. Similarly, the procedure will follow for 0.04 N, 0.06 N, and also for given unknown solution.

Draw a graph between Critical Solution Temperature and concentration of Electrolyte. A straight line will be formed with a positive slope.

Report:

The Concentration of unknown NaCl solution is 0.02 N. On addition of NaCl, the Critical Solution Temperature of Phenol – Water System increases.

Calculations:

S.No.	NaCl Solution	Volume of Water Temperature (T_1)	Clearing Temperature (T_2)	Clouding Temperature $T = (T_1+T_2)/2$	Miscibility Temperature
1	0.02 N	5 mL	5 mL		
2	0.04 N	5 mL	5 mL		
3	0.06 N	5 mL	5 mL		
4	0.08 N	5 mL	5 mL		
5	0.1	5 mL	5 mL		
6	X N	5 mL	5 mL		
7	Y N	5 mL	5 mL		

From graph:

The concentration of unknown NaCl Solution (X)= _____ N.

The concentration of unknown NaCl Solution (Y)= _____ N.

For determining the effect of electrolyte on CST :