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Note : Remove "Table of Content" before including in CP Book

18CHEL26 : ENGINEERING CHEMISTRY LAB

A. LABORATORY INFORMATION

1. Lab Overview

Degree:	B.E	*Program:*	BS
Year / Semester :	2019/1	*Academic Year:*	2019-20
Course Title:	Engineering Chemistry Lab	*Course Code:*	18CHEL26
Credit / L-T-P:	1 / 0-0-2	*SEE Duration:*	180 Minutes
Total Contact Hours:	42 Hrs	*SEE Marks:*	60 Marks
CIA Marks:	40	*Test*	2
Course Plan Author:	Dr. Manju M	*Sign*	Dt : 04-01-2019
Checked By:	Dr. Shankara B.S	*Sign*	Dt : 14-08-2019

2. Lab Content

Unit	Title of the Experiments	Lab Hours	Concept	Blooms Level
	PART- A			
1	Potentiometric estimation of FAS using standard K 2 Cr 2 O 7 solution.	2	Redox Reactions	L4 Analyzing & L5 Evaluation
2	Conductometric estimation of acid mixture.	2	Acid Base Reaction	L4 Analyzing & L5 Evaluation
3	Determination of Viscosity co-efficient of the given liquid using Ostwald's viscometer.	2	Cohesive Force	L4 Analyzing & L5 Evaluation
4	Colorimetric estimation of Copper.	2	Measurement of Optical Density	L4 Analyzing & L5 Evaluation
5	Determination of pKa of the given weak acid using pH meter.	2	PH measurement	L4 Analyzing & L5 Evaluation
6	Flame photometric estimation of sodium and potassium.	2	Atomization	L4 Analyzing & L5 Evaluation
	PART- B			
1	Estimation of Total hardness of water by EDTA method.	2	Complexometric titration	L4 Analyzing &

				L5 Evaluation
2	Estimation of CaO in cement solution by rapid EDTA method.	2	Complexo metric titration	L4 Analyzing & L5 Evaluation
3	Determination of percentage of Copper in brass using standard sodium thiosulphate solution.	2	Iodometri c titration	L4 Analyzing & L5 Evaluation
4	Determination of COD of waste water.	2	Redox titration	L4 Analyzing & L5 Evaluation
5	Estimation of Iron in haematite ore solution using standard K 2 Cr 2 O 7 solution by external indicator method.	2	Redox titration	L4 Analyzing & L5 Evaluatio
6	Estimation of percentage of available chlorine in the given sample of bleaching powder	2	Iodometri c titration	L4 Analyzing & L5 Evaluation

3. Lab Material

Unit	Details	Available
1	**Text books**	
i	Textbook of Engineering Chemistry with Lab Manual 9th Edition (English, Paperback, Shashi Chawla)	In Lib
ii	Vogel's Textbook of Practical Organic Chemistry (5th Edition) 5th Edition by A.I. Vogel (Author), A.R. Tatchell (Author), B.S. Furnis (Author), A.J. Hannaford (Author), P.W.G. Smith (Author)	In Lib
2	Reference books	
i	G.H.Jeffery, J.Bassett, J.Mendham, R.C.Denney, "Vogel's Tex book of quantitative Chemical Analysis Fifth Edition(New) ,	In Lib
ii	O.P.Vermani & Narula, "Theory and Practice in Applied Chemistry", New Age International Publisers.	In Lib
iii	Gary D. Christian, "Analytical chemistry ", 6th Edition, Wiley India.	In Lib
ii	Engineering Chemistry Lab manual	In dept
3	Others (Web, Video, Simulation, Notes etc.)	
i	https://sites.google.com/...chemistry-laboratory-w.	Available on web
ii	https://science.nrao.edu › Facilities › CDL	Available on web
iii	https://www.acs.org/.../chemistryclubs/.../simulati..	Available on web
iv	https://www.augusta.edu/.../chemistryandphysics/	Available on web
v	www.ncl-india.org/	Available on web

4. Lab Prerequisites:

-	-	Base Course:		-	-
SNo	Course Code	Course Name	Topic / Description	Sem	Remarks
1	18CHEL26	Engineering Chemistry Lab	Titrations/students have done these kind of experiments in lower standards.	1	

			Instrumental analysis/students have studied in theory part regarding these experiments.	1	

Note: If prerequisites are not taught earlier, GAP in curriculum needs to be addressed. Include in Remarks and implement in B.5.

5. General Instructions

SNo	Instructions	Remarks
1	Never work in the laboratory unless a demonstrator or teaching assistant is present.	
2	Do not throw waste such as match stems filter papers etc. into the sink. They must be thrown into the waste jars.	
3	Keep the water and gas taps closed expect when these utilities are needed.	
4	Never taste any chemical unless instructed to do so and don't allow chemicals to come in contact with your skin.	
5	While working with gases, conduct the experiment in a fume hood.	
6	Keep all the doors and windows open while working in the laboratory.	
7	You should know about the hazards and properties of every chemical which you are going to use for the experiment. Many chemicals encountered in analysis are poisonous and must be carefully handled.	
8	Sulphuric acid must be diluted only when it is cold .This should be done by adding it slowly to cold water with stirring ,and not vice versa.	
9	Reagent bottles must never be allowed to accumulate on the work bench. They should be placed back in the shelves as and when used.	
10	Containers in which reaction to be performed a little later should be labeled. Working space should be cleaned immediately.	

6. Lab Specific Instructions

SNo	Specific Instructions	Remarks
	Chemical Splash Goggles:	
1	Purchase a pair of chemical safety goggles).	
2	Bring your goggles with you for all laboratory sessions of your chemistry class. You will not be allowed to work in the lab without your goggles	
3	Wear your goggles when anyone in the lab is conducting an experiment.	
	Laboratory Coats:	
4	Purchase a lab coat that fits you well. Lab coats that are too tight or too loose are not safe. Sleeves that are too long should be rolled up.	
5	If your lab coat has not been contaminated with a hazardous substance, you may wash it as you do your other clothing.	
6	If your lab coat becomes contaminated with a hazardous substance, as with any other lab spill, notify your instructor immediately.	
7	Contaminated lab coats will be handled by your instructor as they deem appropriate.	
	Nitrile Gloves:	
8	Nitrile gloves are to be worn only during portions of experiments where specified by the experimental procedure, when instructed by the instructor or supervisor, or when working with substances for which the protocol requires the use of gloves.	
9	Note that nitrile gloves are flammable and will stick to your skin if they burn. Do not wear gloves while working with Bunsen burners.	
10	Do not wear gloves outside the lab. When a chemical comes in contact with a glove, remove the glove immediately and place it in the glove waste.	
11	Do not touch surfaces such as door knobs, computer keyboards, and chairs while wearing Pag gloves.	

12	Gloves with holes or tears must be removed immediately and disposed of properly.	
13	Dispose of gloves at the end of each experiment in the glove waste containers provided in each lab.	

B. OBE PARAMETERS

1. Lab / Course Outcomes

#	COs	Teach. Hours	Concept	Instr Method	Assessment Method	Blooms' Level
			PART- A			
1	Handling different types of instruments for quantitative analysis of samples.	21	Instrumental method of analysis	Demonstrate	Test	L3
			PART- B			
2	Volumetric analysis of various samples quantitatively.	21	Volumetric analysis	Demonstrate	Test	L3
-	**Total**	**42**	-	-	-	-

Note: Identify a max of 2 Concepts per unit. Write 1 CO per concept.

2. Lab Applications

SNo	Application Area	CO	Level
	PART- A		
1	Potentiometric estimation of FAS using standard K 2 Cr 2 O 7 solution.	CO1	L3 & L4
2	Conductometric estimation of acid mixture.	CO1	L3 & L4
3	Determination of percentage of Copper in brass using standard sodium thiosulphate solution.	CO2	L3 & L4
4	Determination of COD of waste water.	CO2	L3 & L4

Note: Write 1 or 2 applications per CO.

3. Mapping And Justification

4. Articulation Matrix

(CO – PO MAPPING)

-	Course Outcomes	Program Outcomes												
#	COs	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12	Level
18CHE271.	Estimate amount of FASpotentio metrically through redox titrations.	x	x	x										
18CHE27.2	Calculate amount of acid mixture conducto metrically through neutralization titration.	x	x	x										
18CHE27.3	Compute amount of copper bu measuring absorbence using optical method	x	x	x										
18CHE27.4	Determine Pka Value of given sample using Ph meter.	x	x	x										
18CHE27.5	Estimation of co-efficient of viscosity of given organic liquid using ostwald's method.	x	x	x										

18CHE27.6	Estimate amount of given sample using flame photo metric method.	x	x	x										
18CHE27.7	Estimation of hardness of given sample by using complexometric titrations.	x	x	x										
18CHE27.8	Caluculate the % of CaO in given sample by rapid EDTA method.	x	x	x										
18CHE27.9	Estimate the % of copper in given brass sample by iodometric titration.	x	x	x										L3
18CHE27.10	Calculate the % of iron in given ore solution using external indicator method.	x	x	x										L3
18CHE2711.1	Estimate total oxidisable impurities of given waste water by redox titrations.	x	x	x										
Estimate the % of chlorine in given bleaching powder sample by iodometric method														

5. Curricular Gap and Content

SNo	Gap Topic	Actions Planned	Schedule Planned	Resources Person	PO Mapping
1					
2					
3					
4					
5					

Note: Write Gap topics from A.4 and add others also.

6. Content Beyond Syllabus

SNo	Gap Topic	Actions Planned	Schedule Planned	Resources Person	PO Mapping
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					

Note: Anything not covered above is included here.

C. COURSE ASSESSMENT

1. Course Coverage

Unit	Title	Teachi	No. of question in Exam	CO	Levels

		ng Hours	CIA-1	CIA-2	CIA-3	Asg-1	Asg-2	Asg-3	SEE		
					PART- A						
1	Potentiometric estimation of FAS using standard $K_2Cr_2O_7$ solution.	02	1	-	-	-	-	-	1	CO1	L3 & L4
2	Conductometric estimation of acid mixture.	02	1	-	-	-	-	-	1	CO1	L3 & L4
3	Determination of Viscosity co-efficient of the given liquid using Ostwald's viscometer.	02	1	-	-	-	-	-	1	CO1	L3 & L4
4	Colorimetric estimation of Copper.	02	1	-	-	-	-	-	1	CO1	L3 & L4
5	Determination of pKa of the given weak acid using pH meter.	02	1	-	-	-	-	-	1	CO1	L3 & L4
6	Flame photometric estimation of sodium and potassium.	02	1	-	-	-	-	-	1	CO1	L3 & L4
					PART- B						
1	Estimation of Total hardness of water by EDTA method.	02	-	1	-	-	-	-	1	CO2	L3 & L4
2	Estimation of CaO in cement solution by rapid EDTA method.	02	-	1	-	-	-	-	1	CO2	L3 & L4
3	Determination of percentage of Copper in brass using standard sodium thiosulphate solution.	02	-	1	-	-	-	-	1	CO2	L3 & L4
4	Determination of COD of waste water.	02	-	1	-	-	-	-	1	CO2	L3 & L4
5	Estimation of Iron in haematite ore solution using standard $K_2Cr_2O_7$ solution by external indicator method.	02	-	1	-	-	-	-	1	CO2	L3 & L4
6	Estimation of percentage of available chlorine in the given sample of bleaching powder	02	-	1	-	-	-	-	1	CO2	L3 & L4
-	**Total**	**42**	**7**	**8**	**5**	**5**	**5**	**5**	**20**	**-**	**-**

Note: Write CO based on the theory course.

2. Continuous Internal Assessment (CIA)

Evaluation	Weightage in Marks	CO	Levels
CIA Exam – 1	10	CO1,	L3 & L4
CIA Exam – 2	10	CO2,	L3 & L4
CIA Exam – 3	10	CO1 & CO2,	L3 & L4
Other Activities – define – Slip test			L2, L3, L4 . ..

BSH

Prepared by   Checked by   Approved

Final CIA Marks	**10**	**-**	**-**

-

SNo	Description	Marks
1	Observation and Weekly Laboratory Activities	05 Marks
2	Record Writing	10 Marks for each Expt
3	Internal Exam Assessment	15 Marks
4	Internal Assessment	40 Marks
5	SEE	60 Marks
-	**Total**	**100 Marks**

PART - A

D. EXPERIMENTS

Experiment 01 : Potentiometric estimation of FAS using standard K2Cr2O7 solution.

-	Experiment No.:	1	Marks		Date Planned		Date Conducted	
1	**Title**	**Potentiometric estimation of FAS using standard K 2 Cr 2 O 7 solution.**						
2	**Course Outcomes**	**Estimation of amount of FAS Potentiometrically through redox reaction**						
3	**Aim**	**Potentiometric estimation of FAS using standard K 2 Cr 2 O 7 solution.**						
4	Material/ Equipment Required	➢ Digital Potentio meter ➢ Calomel & Pt-electrodes ➢ 10ml Burette ➢ 100ml beaker ➢ Glass rod.						
5	Theory, Formula, Principle, Concept	**PRINCIPLE:** Redox titrations can be carried out potentiometrically using platinum-calomel electrode combination. For the reaction: Reduced form → Oxidized form + ne$^-$, The potential, E, is given by Nernst equation, $E = E^o + \frac{0.0591}{n} \log \frac{[\text{Oxidized form}]}{[\text{Reduced form}]}$ Where, E° is the standard potential of the system, and [X] represent the molar concentration x. Suppose that, in beaker we have acidified Fe^{2+} solution, and we add slowly $K_2Cr_2O_7$ from a burette, then following reaction takes place. $6Fe^{2+} + Cr_2O_7^{2-} \rightarrow 6Fe^{3+} + 2Cr^{3+}$ Before the equivalence point, the potential is determined by the Fe^{2+}/ Fe^{3+} system. $E = E^o + \frac{0.0591}{n} \log \frac{[Fe^{3+}]}{[Fe^{2+}]} = 0.75\,V + 0.0591 \log \frac{[Fe^{3+}]}{[Fe^{2+}]}$ The potential of the solution will be around 0.75V (since the contribution from the second term is negligible). After the equivalence point, the potential is determined by the $Cr_2O_7^{2-}$ / Cr^{3+} system.						

$$E = E^o + \frac{0.0591}{6}\log\frac{[Cr_2O7_{2-}]}{[Cr^{3+}]} = 1.33V + 0.00985\log\frac{[Cr_2O7^{2-}]}{[Cr_{3+}]}$$

The potential of the solution will be around 1.3V

At the equivalence point, the potential is average potential of both systems.

Thus, there is an abrupt increase in potential of the solution near the end point.

6 Procedure,

Pipette out 25 cm^3 of ferrous ammonium sulfate (FAS) solution into a 100 ml beaker. Add two test tubes full of dilute sulphuric acid. Immerse the platinum - calomel electrodes assembly in the solution. Measure the potential by adding $K_2Cr_2O_7$ solution from the burette in increments of 0.5 cm^3. Stir the mixture by blowing the air for 10 seconds. Measure the potential of the each addition.

Plot a graph of ΔE/ ΔV against volume of $K_2Cr_2O_7$ as shown in the figure. Find the equivalence point from the graph. Knowing the equivalence point, calculate the normality of the FAS solution, and determine the amount of FAS per liter of the solution.

7 Reaction Equation

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

8 Observation Table,

Vol.of $K_2Cr_2O_7$ in cm^3	E in mv	ΔV	ΔE	ΔE/ΔV
0.0				
0.5				
1.0				
1.5				
2.0				
2.5				
3.0				
3.5				
4.0				
4.5				
5.0				

9 Sample Calculations

Calculations:

$(N_1 V_1)$ $K_2Cr_2O_7$ =.$(N_2 V_2)_{FAS}$

Where V_1 = Vol. of $K_2Cr_2O_7$ at the equivalence point (from the graph)

N_1 = Normality of $K_2Cr_2O_7$ solution =........ N (to be given)

V_2 = Vol. of FAS solution = 25 cm^3

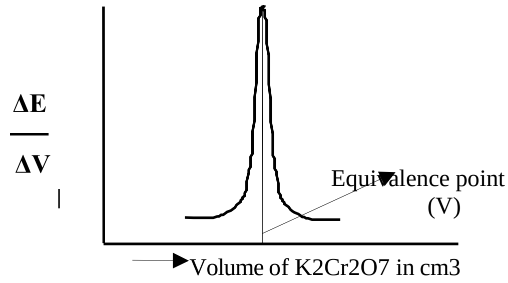
N_2 = Normality of FAS solution = ...

$$\therefore N_2 = \frac{V_1 X N_1}{V_2} = \frac{X}{25} = \text{.............................} N$$

W k t . Mass per dm^3 = Normality x Gram equivalent mass

Mass of FAS per dm^3 = Normality of FAS x Gram equivalent mass of FAS

= ....x 392 g = ... g

10	Graphs, Outputs	
11	Results & Analysis	Normality of FAS = Mass of FAS present in one dm^3 of solution =……. g
12	Application Areas	Application of potentiometry to characterize acid and basic sites in humid substances Testing
		The Techniques to study complexation reactions at the mineral/water.
		Interface No indicator is used; instead the potential is measured across the analyte, typically an electrolyte solution.
13	Remarks	
14	Faculty Signature with Date	

Experiment 02 : Conductometric estimation of acid mixture

-	Experiment No.:	2	Marks		Date Planned		Date Conducted	
1	Title	Conductometric estimation of acid mixture.						
2	Course Outcomes	Calculate amount of acid mixture conductometrically through neutralization reaction						
3	Aim	Conductometric estimation of acid mixture by using standard NaOH(1N).						
4	Material / Equipment Required	➢ Digital Conductometer ➢ Conductivity cell ➢ 10ml Burette ➢ 100ml beaker ➢ Acid mixture ➢ 1N NaOH Solution						
5	Principle	In conductometric titrations, there is a sudden change in conductance of the solution near the neutralization point. However, the change is not sharp and hence the neutralization point is determined graphically by plotting conductivity against titre values. The principle underlying conductometric titrations is the replacement of ions of a particular conductivity by ions of different conductivity during titration. When a mixture of HCl and CH_3COOH is titrated against sodium hydroxide the strong acid, HCl will be neutralized first. The neutralization of the weak acid (CH_3COOH) commences only after the complete neutralization of the strong acid. $NaOH + HCl \longrightarrow NaCl + H_2O$ $NaOH + CH_3COOH \longrightarrow CH_3COONa + H_2O$ The addition of sodium hydroxide to hydrochloric acid decreases the conductance of the latter because highly mobile H^+ ions are replaced by the less mobile Na^+ ion. This trend continues till all the H^+ ions of HCl are neutralized. On continuing the addition of NaOH, conductance increases slowly due the neutralization of acetic acid. Further addition of NaOH raises the conductance steeply due to the presence of free OH^- ions. A typical titration curve is shown in the model graph.						

6 Procedure

Fill a micro burette with the standard NaOH solution. Pipette out 50 cm^3 of the given acid mixture into a clean 100 cm^3 beaker. Place the conductivity cell in the beaker so that the conductivity cell is completely immersed in the acid mixture. Add 0.5 cm^3 NaOH solution from the burette. Stir the solution gently and record the conductance. Continue the measurement of conductance after each addition of 0.5 cm^3 of NaOH till 10 cm^3. Plot a graph of conductance on Y- axis versus volume of NaOH on X-axis. The conductance titration curve is marked by two breaks; the first one corresponds to the equivalence point of HCl (V_1 cm^3) and the second to that of CH_3COOH (V_2 cm^3). From the graph, find the neutralization points and the volume of NaOH required to neutralize the acids

7 Reaction Equation

$$NaOH + HCl \longrightarrow NaCl + H_2O$$

$$NaOH + CH_3COOH \longrightarrow CH_3COONa + H_2O$$

8 Observation Table, Look-up Table, Output

Vol. of NaOH (cm^3)	Conductance (mS)
0.0	
0.5	
1.0	
1.5	
2.0	
2.5	
3.0	
3.5	
4.0	
4.5	
5.0	

9 Sample Calculations

Normality of NaOH = ...N (to be given)

Volume of NaOH required to neutralize HCl = V_1 cm^3

Volume of NaOH required to neutralize CH_3COOH = $(V_2 - V_1)$ cm^3

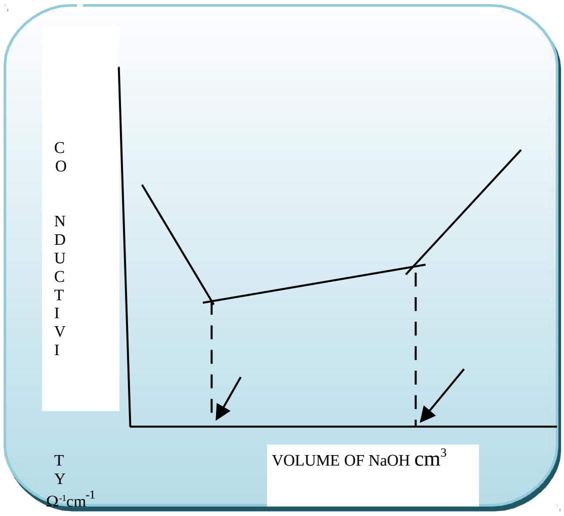
$$N_{HCl} = \frac{[N \times V]_{NaOH}}{50} = \frac{\ldots\ldots \times V_1}{50} = \ldots\ldots\ldots\ldots\ldots\ldots = \text{.................................} \; (a)$$

$$N_{CH_3COOH} = \frac{[N \times (V_2 - V_1)]_{NaOH}}{50} = \ldots\ldots\ldots\ldots\ldots = \text{..........................} (b)$$

Therefore,

weight of HCl/dm^3 = N_{HCl} x Eq.mass of HCl= 'a' x 36.5 = ………….

weight of CH_3COOH/dm^3 = N_{CH_3COOH} x Eq.mass of CH_3COOH = 'b' x 6

10	Graphs, Outputs	 V1 V2
1	Results & Analysis	1) Normality of HCl = ...N 2) Weight of HCl per liter =........ g 3) Normality of CH_3COOH =....... N 4) Weight of CH_3COOH per liter =...... g
12	Application Areas	The experimental determinations of the conducting properties of electrolytic solutions are very important as they can be used to study quantitative behavior of ions in solution.
		They can also be used to determine the many physical quantities such as degree of dissociation and dissociation constants of weak acids and bases, ionic product of water, solubilityand solubility
13	Remarks	
14	Faculty Signature with Date	

Experiment 03 : Determination of Viscosity co-efficient of the given Organic liquid

-	Experiment No.:	3	Marks		Date Planned		Date Conducted	
1	Title	Determination of Viscosity co-efficient of the given liquid using Ostwald's viscometer.						
2	Course Outcomes	Estimation of co-efficient of viscosity of given organic liquid using Ostwald's method.						
3	Aim	Determination of Viscosity co-efficient of the given liquid using Ostwald's viscometer.						
4	Material / Equipment Required	➢ **OSTWALD'S VISCOMETER** ➢ **10ml graduated Pipette** ➢ **Organic Liquids** ➢ **water bath**						
5	Theory	Viscosity arises due to frication between moving layers of a liquid. A liquid flowing through a cylindrical tube of uniform diameter is expected to move in the form of molecular layers. Layer close to the surface is almost stationary while that t the axis of the tube moves faster than any other intermediate layer. A slow moving layer excerts a drag or friction on its nearest moving layer backwards. This property of the liquid, which retards or opposes the motion between the layers, is called viscosity. The Coefficient of viscosity is defined as the tangential force per unit area required maintaining a unit velocity gradient between the two successive layers of the liquid situated unit distance apart. The						

Coefficient of viscosity of a liquid is given by the Poiseuille's formula.

$$\eta = \frac{\pi\, \mathrm{pr}^4\, t}{8\mathrm{Vl}}$$

Where 'v' is the volume of the liquid, 'r' is the radius of the tube and 'p' is the pressure between the two ends of the tube is the Coefficient of viscosity. If equal volumes of the two different liquids are allowed to flow through the same tube under identical conditions then,

$$\frac{\eta_1}{\eta_2} = \frac{t_1 d_1}{t_2 d_2}$$

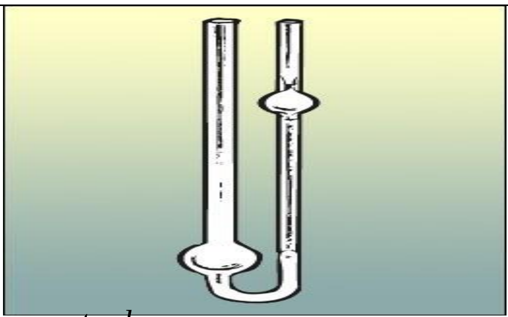
The time't' taken by the given liquid to travel through a certain distance in the tube is determined. The time't' taken by standard liquid to travel through the same distance is measured. Knowing the densities of the two liquids (d_1 and d_2) and also the coefficient of viscosity of the standard liquid, coefficient of viscosity of test liquid is calculated.

6 Procedure

Take a dry viscometer. (Do not wash!) Attach a rubber to the narrow limb. Immerse the viscometer in water bath and fix it vertically to a stand. Transfer 15 cm^3 of the given liquid into the wider limb of the viscometer using a pipette.
Suck the liquid and fill the bulb on the narrow limb slightly above the upper mark. Allow the liquid to flow down through the capillary. Start a stop clock when the level of the liquid crosses the lower mark. Note down the time of flow.
Remove the viscometer from the stand. Remove the rubber tube. Pour out the liquid from the viscometer into the beaker. Using acetone (through a dropper) rinse the viscometer. Dry it in air over for 20 minutes.
Take out the viscometer and fallow a similar procedure for determining the average time of flow for deionized water.(Use a different pipette for water).
Using a thermometer note the temperature of the water bath. This is lab temperature.From your teacher, get the values of d_1 (density of organic liquid), d_2 (density of water) and η_2(Viscosity coefficient of water)
Find η_1(viscosity coefficient of organic liquid) using the relation.

$$\eta_1 = \frac{t_1 d_1}{t_2 d_2} \mathrm{X} \eta_2$$

7 Model Diagram



$$\eta = \frac{\pi\, \mathrm{pr}^4\, t}{8\mathrm{Vl}}$$

$$\eta_1 = \frac{t_1 d_1}{t_w d_w} \mathrm{X}\eta_w = \frac{\qquad \times \qquad}{¿} \quad \frac{\times}{}$$

= ………………….milli poise

8 Observation Table, Look-up Table, Output

OBSERVATION AND CALCULATION:

	Time of flow in seconds			
	Trial 1	Trial 2	Trial 3	Average
Water				
Test liquid				

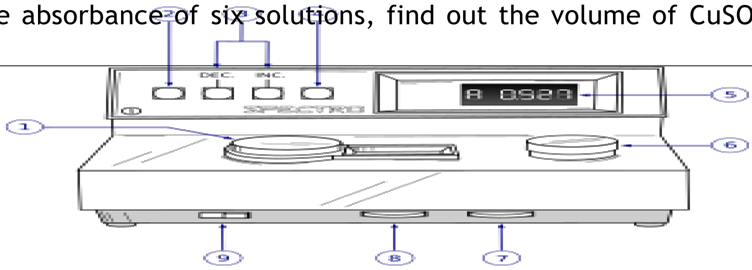
9 Sample Calculations

Lab temperature =……………. °C

		d_1 (density of organic liquid) =………….. g cm^{-3} d_w (density of water) =………….. g cm^{-3} η_w (Viscosity coefficient of water) =………….. millipoise $\frac{\eta_1}{\eta_w} = \frac{t_1 d_1}{t_w d_w}$ $\eta_1 = \frac{t_1 d_1}{t_w d_w} X \eta_w = \frac{\times \quad \times}{¿} = \ldots\ldots\ldots\ldots\ldots\ldots\ldots$ milli poise
10	Graphs, Outputs	Viscosity coefficient of the given liquid
11	Results & Analysis	Viscosity coefficient of the given liquid = ……….. millipoise.
12	Application Areas	**Viscosity** is how **engineers** measure the resistance of fluids to shear stress.
		The **viscosity** equation is useful for calculating a material's **viscosity** when you know the force being applied to the fluid and the resulting velocity
13	Remarks	
14	Faculty Signature with Date	

Experiment 04 : Keywords and identifiers

-	Experiment No.:	4	Marks		Date Planned		Date Conducted	
1	Title	Colorimetric estimation of Copper.						
2	Course Outcomes	Compute the amount of Cu by measuring absorbance using optical method						
3	Aim	Colorimetric estimation of Copper by a givenCuSO4 solution .						
4	Material / Equipment Required	➢ Photo colorimeter ➢ Cuvate tube ➢ 50ml volumetric flask ➢ Copper sulphate solutions ➢ NH3 solutions						
5	Theory, Formula, Principle, Concept	When a monochromatic light of intensity I_o is incident on a colored solution, a part (I_a) of it is absorbed, a part (I_r) is reflected and the remaining part (I_t) is transmitted. Thus, $I_o = I_a + I_r + I_t$ Absorbance is given as $A = \log \frac{I_o}{I_t}$ According to Beer- Lambert's law, A = ЄC l Where, Є = molar extinction coefficient, a constant for any particular colored substance for a given wave length of light, C= Molar concentration of the solution and l = path length. When the path length is kept constant, then A α c. Hence a plot of absorbance, A, against concentration, c, gives a straight line. Chemical analysis through measurements of absorption of light of a particular wavelength is known as colorimetry. The absorbance of light of a particular wavelength by a substance in solution varies directly with its concentration and the thickness of the solution. When the thickness of the medium is kept constant, the absorbance directly depends upon the concentration. A series of solutions with different concentrations of cuprammonium ions is prepared and absorbance of each is measured at **620 nm** radiation. A						

		calibration graph is obtained. The absorbance of cuprammonium ions of unknown solution is also measured and the unknown volume is determined using the calibration graph.
6	Procedure	Take six 50 cm^3 volumetric flasks. Transfer 0, 5, 10, 15 and 20 cm^3 of $CuSO_4$ to first five flasks. Take the unknown solution in the six flasks. Add 5 cm^3 of ammonia solution to each one of the six flasks. Dilute up to the mark and mix well. After 10 minutes, set the absorbance of first solution to zero at **620 nm** radiations in the instrument. Then, measure the absorbance of remaining five solutions with the same settings. Draw a calibration curve by volume of $CuSO_4$ on x-axis and absorbance on y- axis. (Draw a straight line passing through the origin). Using the graph and knowing the absorbance of six solutions, find out the volume of $CuSO_4$ in the sixth flask.
7	Model Diagram	

8 Observation Table, Look-up Table, Output

Sl.No	Vol. of $CuSO_4$ in cm^3	Volume of ammonia sol. in cm^3	Concentration of copper =1.018 mg x vol. of solution	Absorbance
(Blank sol.)	0.0	5.0		
1	5.0	5.0		
2	10.0	5.0		
3	15.0	5.0		
4	20.0	5.0		
5	25.0	5.0		
	Unknown	5.0		

9 Sample Calculations

1000 cm^3 of stock solution contains 4 g of $CuSO_4 . 5H_2O$

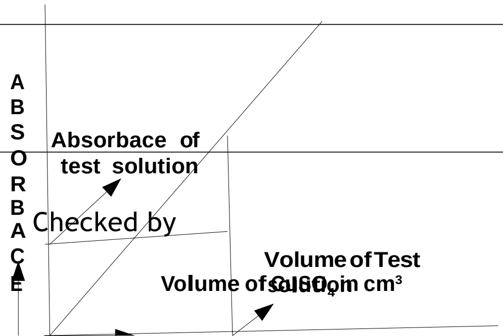
249.54 g of $CuSO_4.5H_2O$ =63.54 g of Cu

4 g of $CuSO_4.5H_2O$ 63.54 × 4 / 249.54 = 1.018 g of Cu per 1000 cm^3 of stock solution

1 cm^3 of $CuSO_4.5H_2O$ 1.018/1000 = 0.001018 g of Cu = 1.018 mg of Cu

Cu present in 'a' cm^3 of test solution = 'a' cm^3 x 1.018 mg =........ mg

10 Graphs



11	Results	**REPORT**: Volume of $CuSO_4$ in the unknown solution = ……… cm^3 Mass of Cu in the unknown solution = …… mg
12	Application Areas	Colorimeters are widely used to monitor the growth of a bacterial or yeast culture.
		Colorimeters are used to measure and monitor the color in various foods and beverages, including vegetable products and sugar.
13	Remarks	
14	Faculty Signature with Date	

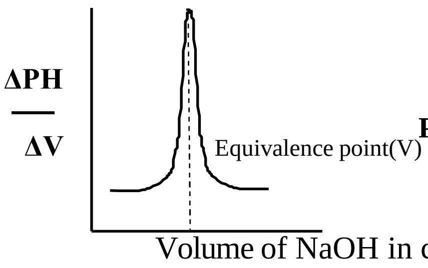
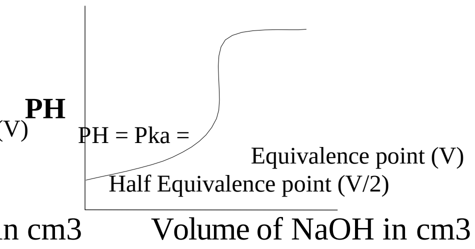
Experiment 05 : Determination of pKa of the given sample using pH meter.

-	Experiment No.:	5	Marks		Date Planned		Date Conducted	
1	Title	Determination of pKa of the given sample using pH meter.						
2	Course Outcomes	Determination of pKa of the given weak acid using pH meter.						
3	Aim	Determination of pKa of the given sample using pH meter.						
4	Material / Equipment Required	• Digital PH-meter • 10ml Burette • 100ml beaker • combining glass electrodes • Weak acid(HCOOH OR CH3COOH) • 1N NaOH Solution • Stirrer • Buffer solutions(pH4, pH7 & pH9)						
5	Theory	A weak acid is an acid, which dissociated partial in solution. Example acetic acid CH_3COOH. When we make a solution of this acid, a part of the acid molecules dissociate. $CH_3COOH \leftrightarrow CH_3COO^- + H^+$ For this reaction, the equilibrium constant, Ka, is given by the equation: $$Ka = \frac{[H^+] X [CH_3COO^-]}{[CH_3COOH]}$$ 'Ka' is also known as acid dissociation constant. The negative logarithm to base 10 of Ka is called pKa. ic., pKa = - $\log_{10}$ Ka. Consider a solution of a weak acid; say acetic acid, in a beaker. Let 'Ka' be the acid dissociation constant. Let us partially neutralized the acid by adding a base, say, NaOH from a burette. Addition of base to the acid result in the formation of salt and water. The pH of the partial neutralized solution is related to pKa of the acid by the Henderson-Hasselbalcs equation, $$pH = pKa + \log\frac{[Salt]}{[Acid]}$$ If we titrate the acid against NaOH, the pH of the mixture in the beaker							

		continuously changes. When we plot a graph of pH vs. volume of NaOH, we get a 'S' shaped curve. We find that there will be sharp jump in pH at the equivalence point. At half equivalence point, [Salt] = [Acid]. Thus, according to the Henderson equation pH becomes equal to pKa at half equivalence point. PROCEDURE: Pipette out 25 cm^3 of the given weak acid into a 100 cm^3 beaker. Immerse the combined glass electrode into the acid. Connect the electrode terminals to a pH meter. Measure the pH of the acid. Add NaOH solution from a micro burette in increments of 0.5 cm^3. After each addition, stir the solution and measure the pH. (After the jump in the pH, take six more readings). Plot a graph of ΔpH/ΔV against volume of NaOH and determine the equivalence point. Plot another graph pH/ volume of NaOH, and note the pH at half equivalence point (Which is nothing but pKa).
6	Procedure	Transfer 25.0 cm^3 of the given weak acid (acetic acid) into a beaker using a pipette. Immerse a glass electrode - calomel electrode assembly into the acid and connect the cell to a pH meter. Measure the pH of the acid. Fill a micro burette with the base (sodium hydroxide). Now add NaOH in the increments of 0.5cm^3, stir the solution carefully, and measure the pH after 10 seconds. Continue the procedure till the pH shows a tendency to increase rapidly. Take few more readings after that. Tabulate the readings. Plot a graph of ΔpH/ΔV against V and determine the equivalence point V_e. Plot a graph of pH (ordinate) against the volume of sodium hydroxide added (abscissa). Determine the pH at half equivalence point. This gives the pk_a of the acid.
7	**Model Diagram**	
8	Observation Table, Look-up Table, Output	(see table below)
9	Sample Calculations	$pH = pKa + \log\frac{[Salt]}{[Acid]}$, [Salt] = [Acid], pH = pKa

Observation Table

Volume of NaOH added (in cm^3)	**P^H**	**ΔV**	**Δ P^H**	**$\frac{\Delta P^H}{\Delta V}$**
0.0				
0.5				
1.0				
1.5				
2.0				
2.5				
3.0				
3.5				
4.0				
4.5				
5.0				

10	Graphs	 
11	Results	**REPORT**: The pKa of the given acid = ...
12	Application Areas	The measurement of pH is used in medical electronics engineering.
13	Remarks	
14	Faculty Signature with Date	

Experiment 06 : Flame photometric estimation of sodium and potassium.

-	Experiment No.:	6	Marks		Date Planned		Date Conducted	
1	Title	Flame photometric estimation of sodium and potassium.						
2	Course Outcomes	Estimation of amount of given sample using Flame photometric.						
3	Aim	Flame photometric estimation of sodium and potassium.						
4	Material / Equipment Required	• Flame photometer FLAPHO or Eppendorf. • Stock solutions of Na^+ and K^+, c = 1 mg/ml. • 6 numbered 100 ml volumetric flasks. • Glass pipettes: 1, 2, 10 ml. • 50ml Burette • 100ml beaker 						
5	Theory	Flame photometry is an atomic emission technique used for the detection of metals. If a solution containing metallic salts is aspirated into a flame, a vapour, which contains metallic atoms, will be formed. The electrons from the metallic atoms are then excited from ground state (E1) to higher energy state (En) where n= 2 , 3, 4,.....7, by making use of thermal energy of flame. From higher energy states, these electrons will return to the ground state by emitting radiations (En-E1= hγ where n=2,3,4 ..7) which are the characteristic of each element. Na* Excitation Energy ↑↓hγ (emission) Dissociation NaCl(s)-----NaCl (g) -----------------------------Na(g) + Cl (g) Energy K*						

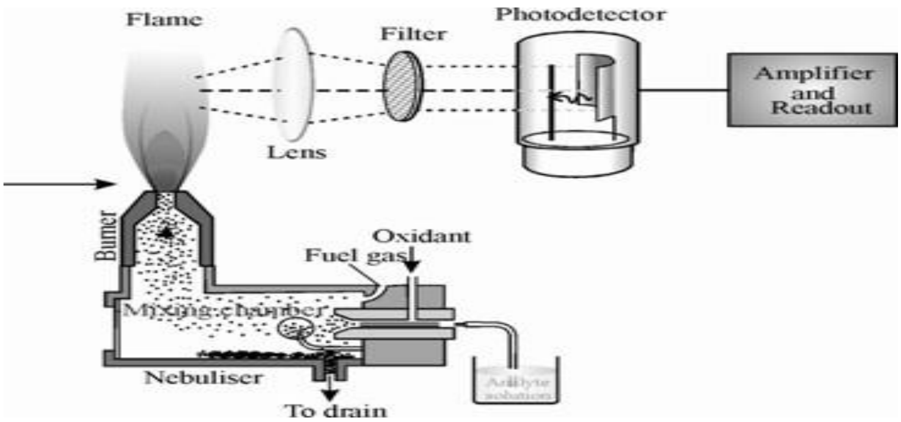
		Excitation Energy ↑↓hγ (emission) Dissociation KCl (s) -----KCl (g) --------------------------------K (g) + Cl (g) Energy Flame photometer correlates the emitted radiations with the concentration of these elements.It is simple and rapid method for the elements that can be easily excited (sodium and other alkali metals). A flame photometer is composed of the pressure regulator and flow meter for fuel gases, an automiser, burner, photosensitive detector and output recorder. A filter of the element whose concentration is to be determined is inserted between the flame and the detector. Propane gas is used as fuel and air or oxygen is used as oxidant. Combination of these two will give a temperature of 1900°C. The whole analysis depends on the flow rate of the fuel, oxidant, the rate of introduction of the sample and droplet size. The sample containing the analyte is aspirated into the flame through automiser. Radiation from resulting flame is collected by the lens and allowed to pass through an optical filter, which permits only the radiation characteristic of the element under investigation into the photocell. The output from the photocell represents the concentration and nature of the element Evaporation Vapourisation Dissociation Thermal excitation M^+X^- MX MX M (gas) + Gas M^+ (g) Flame emission, h●
6	Procedure	Flame photometer uses flammable gases which can ca use explosions if used improperly! Switch the instrument on and off under supervision! Note: Check the flame during work if it goes out, close the gas valve immediately With Eppendorf flame photometer: Transfer 5,10,15,20 and 25 cm^3 of standard sodium chloride solution (which is prepared by weighing accurately 2.542g NaCl into a 1 liter volumetric flask and dissolving the crystals and diluting the solution upto the mark with distilled water and mixing. The solution gives 1ppm /ml) into 100ml standard volumetric flasks and dilute up to the mark with distilled water. Place the distilled water in the suction capillary of the instrument and set the instrument to read zero. Place each of the standard solutions in the suction capillary and set the instrument to read 5,10,15,20 and 25 respectively (rinse with distilled water between each reading). Dilute the given test solution upto the mark, shake well and place the solution in the suction capillary and record the reading. Draw a

calibration curve by plotting the reading (y-axis) and volume of NaCl solution (x-axis). From the calibration curve, find out the volume of the given test solution and from which calculate the amount of Na (58.5 g of NaCl contains 23 g of Na).

Determination of Potassium: Prepare standard solution of potassium and follow the same procedure given above for sodium.

1. Let the instrument warm up for 5-10 minutes.
2. Feed distilled water to the instrument.
3. Select the element Na by turning the selector "Elementwahl".
4. Turn the outer knob "Messbereich" into position "10 0". Pull the "Kompensaton I" knob slightly out and adjust readout to 0. Press the "Kompensation I" knob back. Readjust 0 reading with "Kompensation II" if necessary.
5. Aspirate the most concentrated standard solution (solution number 6) and adjust readout to approximately 350 (on uppermost scale) using inner "Messbereich" knob.
6. Aspirate distilled water – the instrument should read 0.
7. Aspirate standard solutions no. 1, 2, 3, test solution, and then standards 4, 5, 6. Record the results.
8. Repeat 3-7 for solutions of potassium.
9. Aspirate distilled water for at least 5 minutes to clean the system.

7 Model Diagram



8 Observation Table, Look-up Table, Output

Volume of sodium chloride solution (cm^3)	Concentration of Na = 500 x vol / 50 (ppm)	Emission Intensity		Volume of potassium chloride solution (cm^3)	Concentration of K = 500 x vol / 50 (ppm)	Emission Intensity

2.0	20			2.0	20	
4.0	40			4.0	40	
6.0	60			6.0	60	
8.0	80			8.0	80	
10.0	100			10.0	100	
Test solution				Test solution		

9 Sample Calculations

DETERMINATION OF SODIUM:

Weight of Sodium per ml of the solution = 1 mg
1ml of NaCl solution contains 0.002542g of NaCl
58.5 g of NaCl contains 23 g of Na

0.002542 g of NaCl contains = $\frac{23}{58.5} \times 0.002542$

= 1 mg

Therefore 1ml of NaCl solution contains 1 mg of Na
1ml of NaCl solution contains 0.002542g of NaCl
Therefore Xml of NaCl solution contains =
X ×0.002542g of NaCl = ---- ×0.002542g of NaCl
= ------------------------- of NaCl (Y)
Therefore the amount of Na present in above test solution________(Xml) can be calculated by knowing the equivalent weight of Na and molecular weight of NaCl.
Therefore, Y g of NaCl contains

= $\frac{23}{58.5}$ ×Y =-----g= -------- mg

DETERMINATION OF POTASSIUM:

Weight of potassium per ml of the solution = 1 mg
1ml of Kcl solution contains (0.001909g of KCl
74.5 g of KCl contains 39 g of K

= $\frac{39}{74.5}$ ×0.001909 =1 mg

Therefore , 1ml of KCl solution contains 1 mg of K
1ml of KCl solution contains 0.001909g of KCl
Therefore, X ml of KCl solution contains = X × 0.001909g of KCl
=------------ × 0.001909g of KCl

= --------- g of KCl (Y)

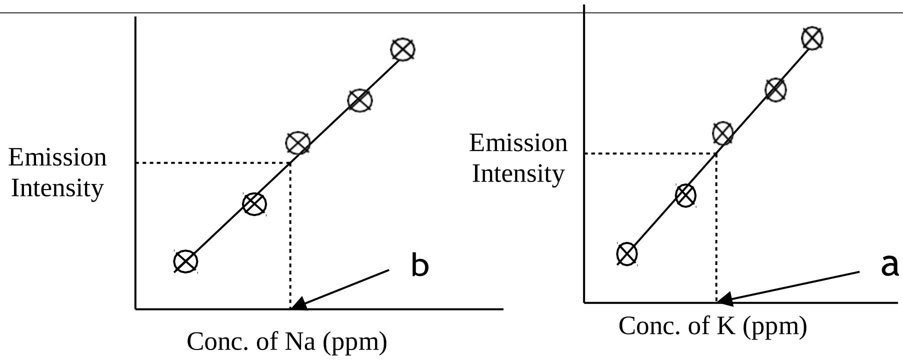
Therefore, the amount of K present in above test solution (X ml) can be calculated by knowing the equivalent weight of K and molecular weight of KCl

Therefore, Y g of KCl contains = $\frac{39}{74.5}$ × Y = ------ g

= ----mg

10 Graphs

Calibration curve



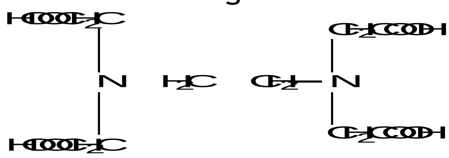
11	Results & Analysis	Result: The weight of Na^+ present in the given test solution = ------------ mg The weight of K+ present in the given test solution= ----------- mg
12	Application Areas	This method is used in determining in ion concentration in BIOLOGICAL FLUIDS in medical electronics engineering.
13	Remarks	
14	Faculty Signature with Date	

PART - B

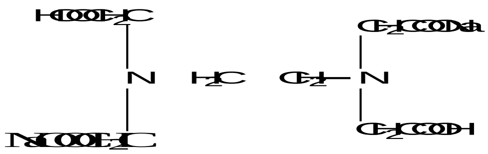
Experiment 01 : Determination of Total hardness of Hard Water sample by using Standard Na2EDTA solution.

-	Experiment No.:	1	Marks		Date Planned		Date Conducted	
1	Title	Determination of Total hardness of Hard Water sample.						
2	Course Outcomes	Estimation of total hardness of given sample of hard water sample using complexometric titration.						
3	Aim	Determination of Total hardness of Hard Water sample by using Standard Na2EDTA solution.						
4	Material / Equipment Required	1. Volumetric flask 2. Burette 3. Pipette 4. Conical flask 5. F annel **Reagents** 1. Na2EDTA Solution 2. Ammonia solutions 3. **Hard water Solution** 4. **NH4-NH4Cl Buffer solution** 5. **EBT Indicator**						
5	Principle	Hardness of water is mainly due to the presence of calcium and magnesium salts in it. Total hardness is the sum of temporary hardness (due to bicarbonates of calcium and Magnesium) and permanent hardness (due to chlorides, sulphates etc., of Calcium and Magnesium). Ethylene diamine tetra acetic acid (EDTA) is a reagent, which reacts with metal ions like Ca^{2+}&Mg^{2+} forming complex compounds. Therefore this reagent can be used to determine the concentration						

of hardness causing substances.



EDTA



Na_2EDTA salt

The completion of the reaction (end point of the titration) is identified using Eriochrome black- T indicator. This is an organic dye, blue in colour. It also forms relatively less stable complexes with bivalent metal ion of Ca &Mg etc., which are wine red in colour. Therefore addition of the indicator to hard water produces wine-red Colour. When EDTA is added to hard water, it first reacts with free metal ions and then attacks the metal-indicator complex .The latter reaction can be represented as

M^{2+}Indicator complex + EDTA→ M^{2+} EDTA complex (COLOURLESS) +free Indicator (Blue) so at the end point a change from wine red to blue colour is Observed. Since the reaction involves the liberation of H^+ ions and the indicator is sensitive to the concentration of H^+ Ions (pH) of the solution a constant Ph of around 10 has to be maintained. For this purpose ammonia-ammonium chloride buffer is used.

6 Procedure

Part-A: Preparation of standard EDTA solution

Weigh the weighing bottle containing disodium salt of Na_2EDTA accurately and transfer the salt in to the funnel placed on a 250 cm^3 volumetric flask. Weigh the bottle again .The difference between the two weights will give the amount of Na_2EDTA transferred. Pour Small quantities of water over the salt on the funnel and transfer the salt in to the Flask. Wash the funnel with the same water 3-4 times; Dissolve the salt by adding 5ml 1:1 Ammonia and make up the solution to the mark and shake well for uniform
Concentration

Part-B: Estimation of hardness of water

Pipette out 25 cm^3 of the given sample of hard water in to a clean conical flask .Add 5 ml of NH_3-NH_4Cl buffer followed by 3-4 drops of Eriochrome black T indicator .Titrate this against Na_2EDTA taken in a burette till the colour changes from wine red to pure blue .Note down the burette reading and repeat the titration to get concordant values.

7 Block, Circuit, Model Diagram, Reaction Equation, Expected Graph

NIL

8 Observation Table, Look-up Table, Output

EDTA in burette	Trial I	Trial 2	Trial 3	Indicator and colour change
Final burette reading				EBT indicator Wine red to clear blue
Initial burette Reading				
Volume of EDTA run down in cm^3				

9 Sample Calculations

OBSERVATION AND CALCULATION:

Part-A: Preparation of Na_2EDTA solution

Weight of the weighing bottle +Na_2EDTA = W_1= g

Weight of the weighing bottle = W_2 = g

Weight of the Na_2EDTA salt transferred = (W1-W2)= g

$$\text{Molarity of EDTA solution} = \frac{\text{Weight of } Na_2EDT\ (W_1 - W_2)\ X\ 4}{\text{Gram molecular wt. of } Na_2 EDTA} = \frac{}{372.}$$

=............M (*a*)

PART-B : Estimation of hardness.

EDTA in burette	Trial I	Trial 2	Trial 3	Indicator and colour change
Final burette reading				EBT indicator Wine red to clear blue
Initial burette Reading				
Volume of EDTA run down in cm^3				

Volume of Na_2EDTA used: *b* cm^3

$1000 cm^3$ of 1M EDTA = 100 g of $CaCO_3$

Therefore *b* cm^3 of *a* molar EDTA = $\frac{bXaX100}{1000}$ = ...(c) g of $CaCO_3$

25 cm^3 of hard water contains = ...(c) g of $CaCO_3$

Therefore $10^6 cm^3$ 0f hard water contains

$$= \frac{c\ X\ 10^6}{25} = {-}{-}{-}{-}{-}{-}{-}{-}\text{ppm}$$

Total hardness of Water = ... ppm of $CaCO_3$

10	Outputs	Total hardness of Water =....... ppm of $CaCO_3$
11	Results & Analysis	**REPORT:** Total hardness of water = ... ppm of $CaCO_3$
12	Application Areas	➢ Complexomtric titration is an efficient method for determining level of hardness of water
13	Remarks	
14	Faculty Signature with Date	

Experiment 02 : DETERMINATION OF CALCIUM OXIDE IN CEMENT SOLUTION.

-	Experiment No.:	2	Marks		Date Planned		Date Conducted	
1	Title	**DETERMINATION OF CALCIUM OXIDE IN CEMENT SOLUTION**						
2	Course Outcomes	Calculate % of Cao in a given cement sample using rapid EDTA method.						
3	Aim	**DETERMINATION OF CALCIUM OXIDE IN CEMENT SOLUTION BY USING STANDARD Na2EDTA SOLUTION.**						
4	Material / Equipment Required	**Apparatus** 6. Volumetric flask 7. Burette 8. Pipette 9. Conical flask 10. F annel **Reagents** 1. Concentrated Hcl						

		2. Na2EDTA Solution 3. **Cement solution** 4. **Glycerol Solution** 5. **Diethyl amine Solution** 6. 4N NaOH Solution 7. Patton and Reeder's indicator
5	Principle	The major constituents of Portland cement are Silicates of calcium, magnesium, aluminum and iron with a small quantity of oxides of alkali metals The average composition of Portland cement is as follows $CaCO_3$ -63.80%; SiO_2 - 20.7%; Al_2O_3 - 5.6% ; Fe_2O_3 - 2.5%; MgO - 3.75%; TiO_2 - 0.23% ; Na_2O - 0.21%; K_2O - 0.51 %; SO^{3-} - 1.75% Use of Eriochrome black-T as indicator gives the total concentration of $Ca^{2+}$and Mg^{2+} ions, While Patton & Reeder's indicator would allow estimation of only Calcium ions in the presence of Magnesium ions. For this purpose P^H of 12-14 has to be maintained. Additions of Diethylamine &Sodium hydroxide serve the purpose.
6	Procedure	**Part A: Preparation of solution of Disodium salt of Na_2EDTA** Weigh the given disodium salt of Na_2EDTA and transfer on to the funnel placed on a 250 cm^3 volumetric flask. Dissolve by adding small amount of DM water. Make it up to the mark and shake well to get uniform concentration. **Part B: Estimation of CaO** Pipette out 25 cm^3 of given cement solution into a clean conical flask using. Add 5 cm^3 of diethyl amine and 5 cm^3 of 1:1 glycerol. Adjust the pH of the solution by adding 10 cm^3 of 4N sodium hydroxide solution. Add a pinch of Patton & Reeder's indicator. Titrate the solution against EDTA solution taken in the burette until the colour changes from wine red to blue. Note down the burette reading and repeat the titration to get concordant values.
7	Block, Circuit, Model Diagram, Reaction Equation, Expected Graph	NIL
8	Observation Table, Look-up Table, Output	(see table below)

EDTA in burette	Trial I	Trial 2	Trial 3	Indicator and colour change
Final burette reading				Patton and Reeder's indicator Wine red to clear blue
Initial burette reading				
Volume of EDTA run down in cm^3				

9	Sample Calculations	**OBSERVATION AND CALCULATION:** **PART A: Preparation of solution of Disodium salt of Na_2EDTA**
		Weight of theweighingbottle+Na_2EDTA= ....g
		Weight of the weighing bottle = ...g
		Weight of the Na_2EDTAsalt transferred=............. g

Molarity of Na_2EDTA solution =

$$\frac{\text{Weight of } Na_2EDTA \text{ salt X4}}{\text{molecular weight of } Na_2EDTA} = \frac{X\,4 \text{ Gram}}{372.24}$$

=..............................M (*a*)

Part B: Estimation of CaO

EDTA in burette	Trial I	Trial 2	Trial 3	Indicator and colour change
Final burette reading				Patton and Reeder's indicator Wine red to clear blue
Initial burette reading				
Volume of EDTA run down in cm^3				

Weight of cement sample in 25 cm^3 = 0. 09 = W g

Volume of EDTA required to react with 25.0 cm^3 of the cement solution = ...'

1000 cm^3 of 1M EDTA = 56.08 g CaO (Molecular mass of CaO = 56.08)

$$b\ cm^3 \text{ of a M EDTA} = \frac{56.08 \times a \times b}{1000 \times 1} \text{ g of CaO}$$

= ....

= .... 'c 'g of CaO

25 .0 cm^3 of cement solution contains 'c'g of CaO

$$\text{Percentage of CaO in the cement sample} = \frac{c \times 100}{W} = \ldots\ldots\ldots\ldots\ldots\ldots$$

=................

10	Outputs	Percentage of CaO in the cement sample =................
11	Results & Analysis	**REPORT:** Percentage of CaO in the cement sample =................
12	Application Areas	This technique is applicable to determine the quality of cement in civil engineering.
13	Remarks	
14	Faculty Signature with Date	

Experiment 03 : DETERMINATION OF PERCENTAGE OF COPPER IN BRASS

-	Experiment No.:	3	Marks		Date		Date	

					Planned		Conducted	

1	Title	**DETERMINATION OF PERCENTAGE OF COPPER IN BRASS**
2	Course Outcomes	Estimation of percentage of Copper in a given alloy by iodometric method.
3	Aim	**DETERMINATION OF PERCENTAGE OF COPPER IN BRASS BY USING STANDARD Na2S2O3 solution.**
4	Material / Equipment Required	**Apparatus** 1. Volumetric flask 2. Burette 3. Pipette 4. conical flask 5. F annel **Reagents** 1. Concentrated glacial acetic acid 2. Standard sodium thiosulphate solution (0.025N) 3. Potassium iodide 4. NH4OH Solution 5. Starch indicator 6. Brass solution
5	Principle	The chief constituents of brass alloy are copper and zinc. It also contains small quantities s tin, lead and iron. The percentage composition of typical brass is copper 50-90, zinc: 20-40, Tin; 0.6, Lead; 0.2, Iron; 0.1 A solution of brass is made by dissolution of the sample in nitric acid. Boiling with urea destroys oxides of nitrogen. Adding ammonia neutralizes excess acid. The solution is changed to weak acidic medium by adding acetic acid. Potassium iodide is added. Iodine is liberated by the cupric ions. Then the solution is tittered against sodium thiosulphate solution using starch as indicator. The amount of sodium thiosulphate consumed is the measure of the amount of copper present
6	Procedure	**PART A: Preparation of Brass solution:** Weigh exactly the given sample of brass into a clean 250 cm^3 conical flask. Add 3cm^3 of 1:1 nitric acid and boil. Add 2 test tube of Dm water and about 1 g of urea. Boil for about 2 minutes destroy oxides nitrogen. Cool the mixture. **PART -B: estimation of copper in brass solution.** Add 1 test tube of Demineralised water to the solution obtained in part A. Add Ammonium hydroxide drop by drop until a pale blue precipitate is obtained. Dissolve the precipitate by adding 5cm3 of acetic acid and 10cm^3 of 20% KI solution.Titrate the librated iodine against standard sodium thiosulphate solution taken in the burette until the solution becomes PALE YELLOW. Add about 2 cm^3 of freshly prepared starch solution as indicator. Continue the titration by adding sodium thiosulphate solutionStrictly drop by drop until the dark blue coloration disappears, leaving behind white ppt. Repeat PART A and Part B to conduct a duplicate. Calculate the percentage of copper present in brass sample.
7	Reaction Equation	$2Cu^{2+}$ + 4KI ---------- Cu_2I_2 + $4K^+$ + I_2 $2Na_2S_2O_3$ + I_2.................................. + $Na_2S_4O_6$
8	Observation Table, Look-up Table, Output	

Burette readings	Sample- I	Sample-II	Sample-III	Indicator and colour change

BSH

Prepared by   Checked by   Approved

		Final				Starch solution. Disappearance of blue colour
		Initial				
		Volume of Sod. Thiosulphate run down (in cm^3)				

9 Sample Calculations

OBSERVATION AND CALCULATION:

SAMPLES	Sample-1	Sample-2	Sample-3
Weight of the brass transferred	g	g	g

PART -B: Estimation of copper in brass solution.

Burette readings	Sample- I	Sample-II	Sample-III	Indicator and colour change
Final				Starch solution. Disappearance of blue colour
Initial				
Volume of Sod. Thiosulphate run down (in cm^3)				

SAMPLE 1:

Normality of Sodium. Thiosulphate = ... (a) N

Volume of the Sod. Thiosulphate = ...(b) cm^3

1000 cm^3 of sod. thiosulphate = 63.54g of copper

∴ b cm^3 of a normal sod.thiosulphate = $\frac{63.54X\ bX\ a}{1000} = \frac{63.54\ X\ \ \ X}{1000}$ g of copper

= .... (c) g of copper

Weight of the brass taken = ... (w) g

(w) g of brass contains (c) g of copper

Therefore, 100g of brass contains = $\frac{cx100}{w} = \frac{\ \ X\ 100}{\ } =$ .... g of copper

(Note : Similarly do the calculation for II and III trial)

10	Outputs	Percentage of copper in brass sample =....................
11	Results & Analysis	Percentage of copper in brass sample =....................
12	Application Areas	This method is used to determine composition of metals in an alloys.
13	Remarks	
14	Faculty Signature with Date	

Experiment 04 : DETERMINATION OF PERCENTAGE OF IRON IN HAEMATITE ORE SOLUTION

-	Experiment No.:	4	Marks		Date Planned		Date Conducted	
1	Title	DETERMINATION OF PERCENTAGE OF IRON IN HAEMATITE ORE						

2	Course Outcomes	Calculate % of Fe in a given ore solution using external indicator method.
3	Aim	**DETERMINATION OF PERCENTAGE OF IRON IN HAEMATITE ORE SOLUTION BY USING STANDARD K2Cr2O7 SOLUTION.**
4	Material / Equipment Required	**Apparatus** 11. Volumetric flask 12. Burette 13. Pipette 14. Conical flask 15. Funnel **Reagents** 1. Concentrated HCl 2. **Haematite ore solution** 3. **SnCl2 Solution** 4. **HgCl2 Solution** 5. Potassium dichromate 6. $[K_3(Fe(CN)_6]$(external)
5	Principle	Haematite is an important ore of iron containing mainly Fe_2O_3 and silica. Estimation of involves the dissolution of the ore in Hydrochloric acid, reducing the Ferric (Fe^{3+}) ions in the solution to Ferrous ($Fe^{2+)}$ ions using a reducing agent like Stannous chloride and the estimation of ferrous ions so obtained by titrating against an Oxidizing agent like Potassium dichromate
6	Procedure	**Part A - Preparation of Potassium Dichromate solution:** Weigh accurately the given potassium dichromate crystals and transfer on to the funnel placed on a 250 cm^3 volumetric flask. Dissolve by adding small quantities of DM water and make upto mark. Shake well to get uniform concentration. **Part B Estimation of Iron:** Pipette out 25 cm^3 of the given Haematite solution in to a clean conical flask. Add 5 cm^3 of concentrated Hydrochloric acid. Heat the solution nearly to boiling. Add Stannous chloride drop by drop to the HOT solution until the solution becomes Colureless. Add 2-3 drops of stannous chloride in excess. Cool the solution to room temperature. Add 2 test tube of DM water followed by 5 cm^3 of Mercuric Chloride at a strech. A silky White precipate is formed. Reject the contents of the flask and repeat The experiment if NO PRECIPATE or GREYISH ppt is formed. Titrate the solution against standard potassium dichromate solution taken in the burette using potassium ferricyanide as an EXTERNAL INDICATOR. In the beginning take out a drop of the reaction mixture using a clean glass rod and mix it with a drop of the indicator arranged on a paraffin paper. The colour of the drop of indicator changes to blue. Take out a drop of the reaction mixture after every addition of $K_2Cr_20_7$ and mix it with a fresh drop of the indicator. appearance of blue or green colour indicates that the END point is not reached. At the end point a drop of the reaction mixture fails to give either blue or green coloration. Note down the burette reading and repeat the experiment for agreeing values.
7	Reaction Equation	$2FeCl_3 + SnCl_2 \rightarrow 2FeCl_2 + SnCl_4$ Yellow Colorless $SnCl_2 + 2HgCl_2 \rightarrow SnCl_4 + Hg_2Cl_2$ Silky white $K_2Cr_2O_7 + 8\ HCl \rightarrow 2KCl + 2\ CrCl_3 + 4H_2O + 3[O]$ $\underline{(2FeCl_2 + 2\ HCl + [O] \rightarrow 2\ FeCl_3 + H_2O)\ X\ 3}$ $K_2Cr_2O_7 + 14\ HCl + 6FeCl_2 \rightarrow 2KCl + 6FeCl_3 + 2CrCl_3 + 7\ H_2O$ Green

8 Observation Table, Look-up Table, Output

Burette readings	**Trail I**	**Trail II**	**Trail III**	Indicator and colour change
Final burette reading				$[K_3(Fe(CN)_6]$(external) Blue to no change in the colour of indicator.
Initial burette reading				
Volume of $K_2Cr_2O_7$ run down (in cm^3)				

9 Sample Calculations

PART A: Preparation of potassium dichromate solution

Weight of the weighing bottle + $K_2Cr_2O_7$ = g

Weight of the weighing bottle = g

Weight of the $K_2Cr_2O_7$ salt transferred = g

$$\text{Normality of } K_2Cr_2O_7 \text{ solution} = \frac{\text{Wt. of } K_2Cr_2O_7 X\ 4}{\text{Gram Eq . wt . of } K_2Cr_2O_7} = \frac{X}{49.06}$$

Part B: Estimation of Iron:

Burette readings	**Trail I**	**Trail II**	**Trail III**	Indicator and colour change
Final burette reading				$[K_3(Fe(CN)_6]$(external) Blue to no change in the colour of indicator.
Initial burette reading				
Volume of $K_2Cr_2O_7$ run down (in cm^3)				

Volume of $K_2Cr_2O_7$ consumed: (***b***) cm^3

Weight of haematite ore dissolved in 250 cm^3 of the solution = 1.025 g

1000 cm^3 of 1N$K_2Cr_2O_7$ = 1 equivalent of iron

= 55.85 g of Iron

Therefore (***b***) cm^3 of (***a***) normal $K_2Cr_2O_7$ =

$$\frac{55.85\ X\ b\ X\ a}{1000} = \frac{55.85\ X \quad X}{1000}$$

= ..... (c) g of iron

25 cm^3 of haematite ore solution contains (c) g of iron

250 cm^3 of haematite ore solution contains 10 X (c) =------------------- (d) g of iron

Therefore, 100g of haematite ore contains = $\frac{dx100}{1.025} = \frac{X\ 100}{1.025}$ = ---------------

Percentage of iron in given haematite ore sample = ...

10	Outputs	Percentage of iron in given sample of hematite = ...
11	Results & Analysis	**REPORT**: Percentage of iron in given sample of hematite.
12	Application Areas	This method is used to determine composition of metals in its ore in metallurgical process.
13	Remarks	
14	Faculty Signature with Date	

Experiment 05 : DETERMINATION OF CHEMICAL OXYGEN DEMAND (COD) OF WATER

-	Experiment No.:	5	Marks		Date Planned		Date Conducted	
1	Title	DETERMINATION OF CHEMICAL OXYGEN DEMAND (COD) OF WATER						
2	Course Outcomes	Estimation of total oxidizable impurities present in sewage water through redox titration.						
3	Aim	DETERMINATION OF CHEMICAL OXYGEN DEMAND (COD) OF INDUSTRIAL WAST WATER SAMPLE BY USING STANDARD FAS SOLUTION.						
4	Material / Equipment Required	**Apparatus** 16. Volumetric flask 17. Burette 18. Pipette 19. Conical flask 20. F anal **Reagents** I. Concentrated H2SO4 II. Ferrous ammonium sulphate (FAS) III. Potassium dichromate IV. Ferroin indicator V. Wast water sample						
5	Principle	COD is a measure of oxygen equivalent of that portion of oxidisable materials that can be oxidized by a strong oxidizing agent. Chemical oxygen demand is an important parameter in industrial wastewater treatment. Straight chain aliphatic compounds, aromatic hydrocarbons, straight chain alcohol, acids, pyridine and other oxdisable material are present as impurities in wastewater. Straight chain compounds, acetic acid etc. are oxidisabe more effectively when silver sulphate is added as catalyst. Addition of mercuric sulphate would help avoid interference of chloride ions.						
6	Procedure	**Part A- Preparation of standard ferrous ammonium sulphate (FAS) solution**: Weigh accurately the given FAS and transfer it into a 250 cm^3 standard flask using a funnel. Add 30cm^3 of dilute sulphuric acid followed by about 100 cm^3 of water. Dissolved, make it up to the mark and shake well for uniform concentration. **Part-B: Blank titration**:						

Pipette out 25cm^3 of potassium dichromate into a conical flask-using pipette. Add 10 cm^3 of 1:1 sulphuric acid containing mercuric sulphate and silver sulphate and 3 drops ferroin indicator. Titrate against FAS taken in the burette until the colour changes from blue green to reddish brown. Note the burette reading and repeat the titration to get concordant values.

Part-C: Back titration:

Pipette out 25 cm^3 of given sample of wastewater into a conical flask. Add 25 cm^3 of standard potassium dichromate solution using a pipette. Add 10 cm3 of 1:1 sulphuric acid containing mercuric sulphate and silver sulphate while shaking the flask constantly. Reflux the content of flask for 30 minutes. Cool to room temperature. Add 3-4 drops ferroin indicator and Titrate against FAS solution taken in the burette until the colour changes from bluish green to reddish brown. Note down the burette reading and repeat the titration to get concordant values.

7 Reaction Equation

8 Observation Table, Look-up Table, Output

Burette readings	Trail I	Trail II	Trail III	Indicator and colour change
Final burette reading				Ferroin indicator Blue green to Reddish brown
Initial burette reading				
Volume of FAS run down (in cm^3)				

9 Sample Calculations

OBSERVATION AND CALCULATION:

PART A: Preparation of Ferrous ammonium sulphate (FAS) solution:

Weight of the weighing bottle + FAS	=	g
Weight of the weighing bottle	=	g
Weight of the FAS salt transferred	=	g

Normality of FAS solution =

$$\frac{\text{Wt. of FAS X4}}{\text{Gram eq. wt. of FAS}} = \frac{X\ 4}{392} = \ldots\ldots\ldots\ldots\ldots N(a)$$

Volume of FAS consumed in the blank titration =........(**b**) cm^3

Part-B: Back titration:

Burette readings	Trail I	Trail II	Trail III	Indicator and colour change
Final burette reading				Ferroin indicator Blue green to Reddish brown
Initial burette reading				
Volume of FAS run down (in cm^3)				

Back titrate valve = (**c**) cm^3

Amount of potassium dichromate (in terms of FAS) that has reacted with water sample =_ (**b**)-(**c**) cm^3

1000 cm^3 of 1N FAS solution = 1 equivalent of oxygen = 8 g of oxygen.

		***b* -c** cm^3 of '**a**' N FAS solution = $\frac{(b-c) X a X 8}{1000} = \frac{}{1000}$ = ... (**d**) g of oxygen 25 cm^3 of wastewater requires (**d**) g of oxygen Therefore, 1000 cm^3 of waste water requires = $\frac{d x1000}{25}$ =.....g oxygen COD of the given sample of water =...... mg/dm^3 of oxygen_
10	Outputs	COD of the given sample of water =........ mg/dm^3 of oxygen
11	Results & Analysis	**REPORT**: COD of the given sample of water = ...mg/dm^3 of oxygen
12	Application Areas	This technique is used to maintain standard parameters in industrial waste water in environmental engineering.
13	Remarks	
14	Faculty Signature with Date	

Experiment 06 : Estimation of percentage of available chlorine in the given sample of bleaching powder

-	Experiment No.:	6	Marks		Date Planned		Date Conducted	
1	Title	Estimation of percentage of available chlorine in the given sample of bleaching powder						
2	Course Outcomes	Estimation of % of chlorine in a given bleaching powder sample by Iodometric method.						
3	Aim	Estimation of percentage of available chlorine in the given sample of bleaching powder by using Standard Na2S2O3 Solution.						
4	Material / Equipment Required	**Apparatus** I. Mortar and pestle II. Volumetric flask III. Burette IV. Pipette V. Erlenmeyer flask. **Reagents** VI. Concentrated glacial acetic acid VII. Standard sodium thiosulphate solution (0.025N) VIII.Potassium iodide IX. Starch indicator X. Iodine solution (0.025 N).						
5	Principle	Bleaching powder is commonly used as a disinfectant. The chlorine present in the bleaching powder gets reduced with time. So, to find the exact quantity of						

		bleaching powder required, the amount of available chlorine in the sample must be found out. Chlorine will liberate free iodine from potassium iodide solution when its pH is 8 or less. The iodine liberated, which is equivalent to the amount of active chlorine, is titrated with standard sodium thiosulphate solution using starch as indicator.
6	Procedure	1. Dissolve 1g bleaching powder in 1 litre of distilled water in a volumetric flask, and stopper the container. (This can be done by first making a paste of the bleaching powder with mortar and pestle.) 2. Place 5 mL acetic acid in an Erlenmeyer flask and add about 1g potassium iodide crystals. Pour 25 mL of bleaching powder solution prepared above and mix with a stirring rod. 3. Titrate with 0.025 N sodium thiosulphate solution until a pale yellow colour is obtained. (Deep yellow changes to pale yellow.) 4. Add 1mL of starch solution and titrate until the blue colour disappears. 5. Note down the volume of sodium thiosulphate solution added (V_1). 6. Take a volume of distilled water corresponding to the sample used. 7. Add 5 mL acetic acid, 1g potassium iodide and 1 mL starch solution. 8. If blue colour occurs, titrate with 0.025 N sodium thiosulphate solution until the blue colour disappears. 9. Record the volume of sodium thiosulphate solution added (A_1). 10. If no blue colour occurs, titrate with 0.025 N iodine solution until a blue colour appears. Note down the volume of iodine (A_2). 11. Then, titrate with 0.025 N sodium thiosulphate solution till the blue colour disappears. Record the volume of sodium thiosulphate solution added (A_3). Note down the difference between the volume of iodine solution and sodium thiosulphate as A_4(A_4=A_2- A_3). Note: Blank titration is necessary to take care of the oxidising or reducing reagents' impurities.
7	Reaction Equation	A_4(A_4=A_2- A_3).
8	Observation Table, Look-up Table, Output	Bleaching powder solution x Standard sodium thiosulphate solution (0.025 N)

Trail no.	Volume of bleaching Powder solution(mL)	Burette reading		Volume of tit rant(mL)
		Initial	Final	

Distilled water × Standard sodium thiosulphate solution (0.025 N)

Trail no.	Volume of bleaching Powder solution(mL)	Burette reading		Volume of tit rant(mL)
		Initial	Final	

Distilled water x Standard iodine solution (0.025N)

Trail no.	Volume of bleaching Powder solution(mL)	Burette reading		Volume of tit rant(mL)
		Initial	Final	

9 Sample Calculations

$$\text{mg of } Cl_2/\text{mL (B)} = \frac{(V - A_1) \text{ or } (V + A_4) \times N \times 35.46}{\text{mL of bleaching powder solution taken}}$$

1000 mL of bleaching powder solution contains 1000 x B mg of Cl_2

i.e., 1000 mg bleaching powder contains 1000 B mg of Cl_2

therefore, 100 mg of bleaching powder contains $= \frac{1000 \text{ X B}}{10}$

% of chlorine available = ...

10	Outputs	Available chlorine in the given bleaching powder is . %
11	Results & Analysis	Available chlorine in the given bleaching powder is ..%
12	Application Areas	This technique is used to determine the quality of bleaching powder sample.
13	Remarks	
14	Faculty Signature with Date	