MAHARAJA INSTITUTE OF TECHNOLOGY MYSORE

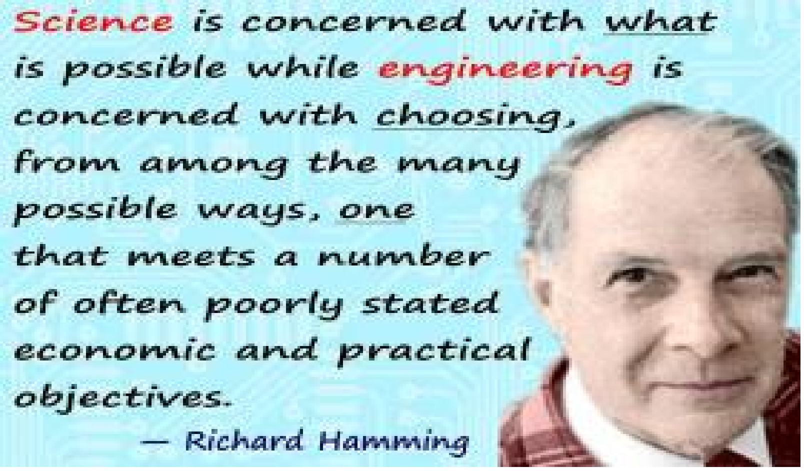
ENGINEERING CHEMISTRY

LABORATORY MANUAL

{As per **Choice Based Credit System (CBCS) Scheme** VTU, Belagavi}

Visvesvaraya Technological University

(I/II SEM, B.E.: Sub. Code: 18 CHEL 16/ 26)



Science is concerned with what is possible while engineering is concerned with choosing, from among the many possible ways, one that meets a number of often poorly stated economic and practical objectives.

— Richard Hamming

Name: ___________________________________

Section: ___________________________________

Batch: ___________________________________

USN: ___________________________________

Department of Chemistry, MIT, Mysore
Belawadi, S. R. Patna Tq, Mandya – 571477

MAHARAJA INSTITUTE OF TECHNOLOGY MYSORE

Vision: *"To be recognized as a premier technical and management institution promoting extensive education fostering research, innovation and entrepreneurial attitude"*

Mission:

- *To empower students with indispensable knowledge through dedicated teaching and collaborative learning.*
- *To advance extensive research in science, engineering and management disciplines.*
- *To facilitate entrepreneurial skills through effective institute-industry collaboration and interaction with alumni.*
- *To instill the need to uphold ethics in every aspect.*
- *To mould holistic individuals capable of contributing to the advancement of the society.*

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Principal

DEPARTMENT OF CHEMISTRY

MAHARAJA INSTITUTE OF TECHNOLOGY MYSORE

VISION:

Foster technologically relevant chemical education in engineering applications to bring about excellence in research and innovation.

MISSION:

- To endow relevant knowledge in chemical science with eminent teaching and demonstration.
- To disseminate greater understanding through research in chemical and related disciplines.
- To facilitate awareness of the impact of chemical attributes on our environment.

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HOD

ENGINEERING CHEMISTRY LABORATORY			
Semester	I/II	CIE MARKS	40
Course Code	18CHEL16/26	SEE Marks	60
Teaching Hours/ week (L: T:P)	0:0:2	Exam Hours	03
CREDITS -01			

Course Objectives:

To provide students with practical knowledge of quantitative analysis of materials by classical and instrumental methods for developing experimental skills in building technical competence.

Instrumental Experiments

1. Potentiometric estimation of FAS using standard $K_2Cr_2O_7$ solution.
2. Conductometric estimation of acid mixture
3. Determination of Viscosity co-efficient of the given liquid using Ostwald's viscometer.
4. Colorimetric estimation of Copper
5. Determination of pKa of weak acid using pH meter
6. Flame Photometric Estimation of Sodium and Potassium

Volumetric Experiments

1. Estimation of Total hardness of water by EDTA complexometric method.
2. Estimation of CaO in cement solution by rapid EDTA method.
3. Determination of percentage of Copper in brass using standard sodium thiosulphate Solution.
4. Determination of COD of waste water.
5. Estimation of Iron in haematite ore solution using standard $K_2Cr_2O_7$ solution by external Indicator method.
6. Estimation percentage of chlorine in the given sample of bleaching powder (Iodometric method).

Course Outcomes:

On completion of this course, students will have the knowledge in,

- Handling different types of instruments for analysis of materials using small quantities of materials involved for quick and accurate results

- Carrying out different types of titration for estimation of concerned in materials using comparatively more quantities of materials involved for good results.

Conduction of Practical Examination:

1. Examination shall be conducted for 100 marks, later reduced to 60 marks.
2. All experiments are to be included for practical examination.
3. One instrumental and another volumetric experiment shall be set.
4. Different experiments shall be set under instrumental and a common experiment under Volumetric.

Reference Books:

1.G.H.Jeffery,J.Bassett,J Mendham and R.C.Denney,”**vogel’s text book of Quantitative chemical analysis**”

2.O.P.Vermani & Narula,”**Theory and practice in Applied chemistry**”,new age international publishers.

3. Gary D.Christian,”**Analytical chemistry**”,6th edition,Wiley India.

CO’s	DESCRIPTION OF THE OUTCOMES
18CHEL16.1/26.1	**Understand and apply** the fundamental of chemical reaction procedural adaptation in practices and safe usage of equipment in laboratory and present the findings appropriately.
18CHEL26.2/26.2	**Apply** the principles related to chemistry to conduct investigation for given scenario under study.
18CHEL16.3/26.3	**Analyze** the key input and outcomes of experiment on the given problem scenario, infer the correctness of the selected parameter based on efficacy of solution and present them suitably.

DO’s and DONT’s:-

DO’s:

- Wear proper uniform with identity cards in the laboratory.
- Should maintain disciplines in the laboratory.
- Handle all the equipment’s and chemicals carefully.
- Follow the safety precautions while performing experiment.
- Enter the lab in-time as per the schedule.
- Comply the instructions given by the faculty and instructor.
- Arrange the chairs/equipments before you leave the lab.
- Keep your surrounding clean and tidy.

DONT’s:

- Do not walk around in the lab unnecessarily.
- Do not go out of the lab without permission.
- Do not eat, smoke or drink in the Laboratory.
- Do not walk bare footed in the Laboratory.
- Never do unauthorized experiment.
- Never taste any chemicals.
- Do not use any equipment or chemicals unless you are trained and approved as a user by your supervisor.

Learn as much as you can while you are young. Since

Life becomes too busy later……

-Dana Stewart Scott

GENERAL INSTRUCTIONS

- Maintain silence in the laboratory.
- Keep your working table clean and tidy.
- Keep the reagents in their proper places after use to avoid misplace and do not contaminate.
- Use Dustbin provided to put waste papers and filter paper.
- Do not pour concentrated acids into the sink. If they are to be poured, flush them using water liberally.
- To turn out to be a better analyst, understand the theory behind the reactions you carry out.
- Use the specified quantities of reagents.
- Do not use wet hand during weighing.
- Neatly written practical record for the experiment performed in the previous class and manual book for the experiment to be performed shall be produced at the beginning of practical class.
- Bring all the necessary things such as calculator, graph sheet, pencil, scale etc., to perform the experiment in stipulated time.
- Confine long hair when in the lab so that it will not catch on fire or come into contact with chemicals.

CONTENTS OF EXPERIMENTS

Part – A

INSTRUMENTAL ANALYSIS

Sl. No.	Date	Title of the Experiments	Marks
1		**Colorimetric** estimation of copper.	
2		Determination of **pKa** of a weak acid using **pH meter**.	
3		**Potentiometric** estimation of FAS using standard $K_2Cr_2O_7$ solution.	
4		**Conductometric** estimation of an Acid mixture using standard NaOH solution.	
5		Determination of viscosity coefficient of a given liquid using Ostwald’s **Viscometer**.	
6		**Flame Photometric** estimation of Sodium and Potassium in the given sample of Water.	

Part – B

VOLUMETRIC ANALYSIS

Sl. No.	Date	Title of the Experiments	Marks
1		Determination of **Total Hardness** of a sample of Water using Disodium salt of EDTA.	
2		Determination of **CaO** in the given sample of Cement by Rapid EDTA method.	
3		Determination of **COD** of the given Industrial Waste water sample.	
4		Determination of Percentage of **Copper** in Brass using standard Sodium thiosuplhate solution.	
5		Estimation **percentage of chlorine** in the given sample of bleaching powder (Iodometric method).	
6		Determination of **Iron** in a given sample of Haematite ore solution using $K_2Cr_2O_7$ crystals by external indicator method.	

INSTRUMENTAL METHODS OF CHEMICAL ANALYSIS

PART - A

OBSERVATIONS AND CALCULATIONS

Volume of NaOH added in ml	p^H	Δp^H	$\Delta p^H / \Delta V$
0.0			
0.5			
1.0			
1.5			
2.0			
2.5			
3.0			
3.5			
4.0			
4.5			
5.0			
5.5			
6.0			
6.5			
7.0			
7.5			
8.0			
8.5			
9.0			
9.5			
10.0			

PART - A

INSTRUMENTAL METHODS OF CHEMICAL ANALYSIS

EXPERIMENT NO. 1 **Date:** ☐☐☐☐☐☐☐☐

DETERMINATION OF pKa OF A WEAK ACID USING pH METER

THEORY:

The strength of an acid is experimentally measured by determining its equilibrium constant or disassociation constant (Ka). Strong acids are completely ionized in aqueous solution and hence it is impossible to determine the disassociation constant of strong electrolytes. But in case of weak electrolytes are partially ionized in aqueous solution and hence the equilibrium constant of weak electrolytes can be experimentally determined.

Let us consider weak acid like formic acid, which ionizes, freely in aqueous solution as,

$$HCOOH\,(aq) + H_2O\,(l) \overset{ka}{\rightleftharpoons} H_3O^+\,(aq) + HCOO^-\,(aq)$$

$$\therefore K_a = \frac{[HCOO^-]\,[H_3O^+]}{[HCOOH]}$$

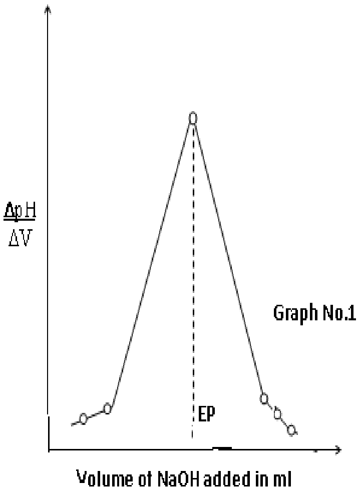
pKa is a modern method of expressing the strength of weak acid ,

ie., $pKa = -\log_{10} Ka$

pKa value is determined by measuring the changes in pH of acid solutions at different amount of the base added. In the titration of an acid with a base, the pH of the solution rises gradually at first and then more rapidly. At the equivalence point there is a very sharp increase in pH for a very small quantity of base added. Beyond the equivalence point, the pH increases slightly on addition of base. The titration curve is obtained by plotting change in pH against different volume of the base added and from the graph, determined the equivalence point of an acid. The pKa value of an acid can be calculated using Henderson equation ,

$$pH = pKa + 1og_{10} \frac{[Salt]}{[Acid]}$$

At half equivalence point [salt] = [acid] and hence pH at half equivalence point gives the pKa value of weak acid, i.e. pH = pKa .

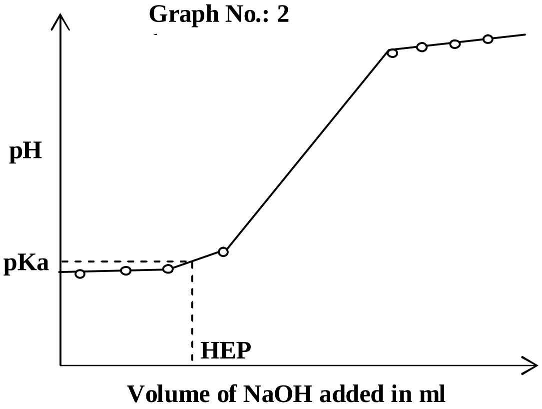


Scale :

X –axis, 1.0 cm = _______ ml of NaOH

Y –axis, 1.0 cm = _______ ΔpH/ΔV

Graph No.: 2



Scale :

X –axis, 1.0 cm = _______ ml of NaOH

Y –axis, 1.0 cm = _______ pH

PROCEDURE:

Make up the given weak acid in a 100 ml standard flask to the mark by adding distilled water and mix well for uniform concentration. Pipette out 25 ml of the given weak acid into a clean 100 ml beaker.

Connect the combined electrode (i.e. glass and calomel electrode) to the pH meter and immerse the electrode in to the acid taken in a beaker. Measure the pH of the acid . Add 0.5 ml of sodium hydroxide solution taken in a micro burette in to the acid, stir well and measure the pH of the acid and continue the addition of sodium hydroxide until the sudden raise in the pH of the acid occur. Then take few more readings and take the difference in the pH of acid (ΔpH) and the volume of sodium hydroxide added (ΔV). Plot the graph, ΔpH/ΔV against the volume of sodium hydroxide added (graph No.1) and from the graph 1, determine the equivalence point of the acid. Plot the graph, pH against the volume of sodium hydroxide added (graph No 2). Determine the half equivalence point that gives the pKa value of weak acid by using the graph 2.

RESULTS:

1. Equivalence Point =_________________ ml of NaOH
2. Half E.P =_________________ ml of NaOH
3. pH at H.E.P = pKa of the given acid =______________

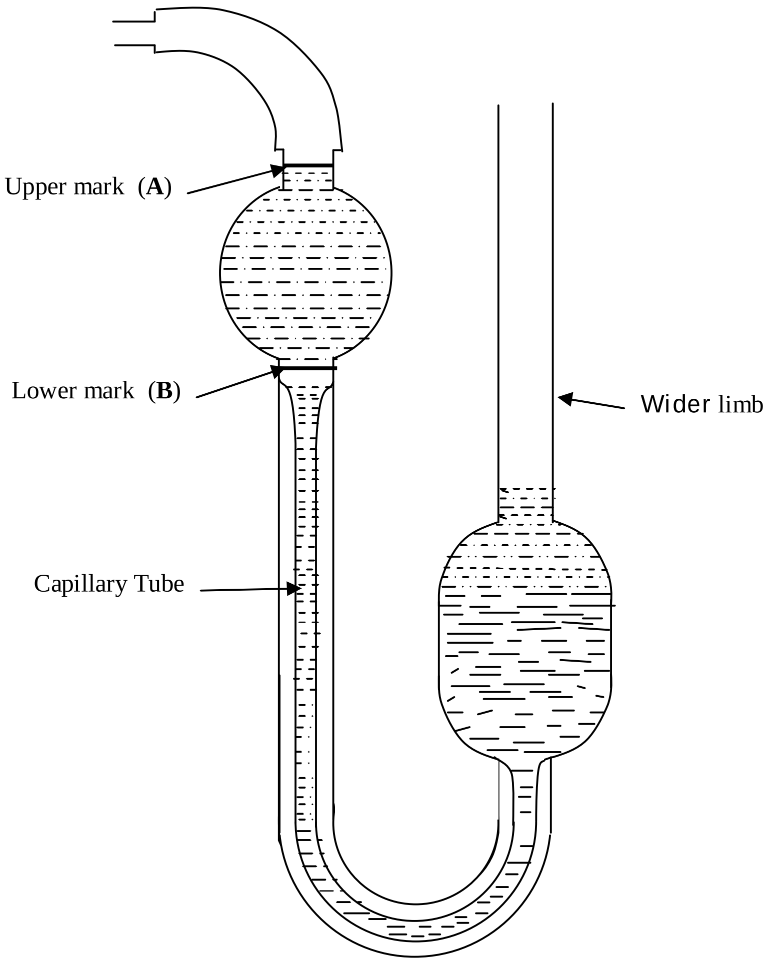


Fig. Viscometer

EXPERIMENT NO.2 **Date:**

DETERMINATION OF VISCOSITY COEFFICIENT OF A GIVEN LIQUID USING OSTWALD'S VISCOMETER

THEORY: Viscosity arises due to internal friction between moving layers of molecules. A liquid flowing through a cylindrical tube of uniform diameter is expected to move in the form of molecular layers. A layer close to the surface is almost stationary while that at the axis of tube moves faster than any other intermediate layer. A slow moving layer exerts a drag or friction on its nearest moving layer backwards. This property of a liquid by which it retards or opposes motion of a liquid 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 any two successive layers of a liquid situated unit distance apart. The coefficient of viscosity of a liquid is given by Poiseuille's formula, $\eta = \frac{\pi P r 4 t}{8 V l}$, Where P = h d g

$$\eta = \frac{\pi h d g r 4 t}{8 V l}$$

Where η is the coefficient of viscosity of the liquid, V = volume of the liquid, r = radius of the tube, 1 = length of the tube, P is the pressure difference between the two ends of the tube, h = height of the capillary tube, d = density of the liquid, and g = gravitational force. If equal volumes of two different liquids are allowed to flow through the same tube under identical conditions, then,

$$\frac{\eta_w}{\eta_L} = \frac{t_w d_w}{t_L d_L}$$

Where, η_w = co – efficient of viscosity of the standard liquid (water), and η_L = co – efficient of viscosity of the given liquid which can be determined. Where, t_w **=** time taken by the standard liquid (Water), t_L = time taken by the given liquid, d_w = density of standard liquid (water), d_L **=** density of given liquid. Viscosity is a temperature dependent property and is inversely proportional to the temperature (T), i.e. **η α 1/ T**. Hence the measurements are carried out at constant temperature.

OBSERVATIONS AND CALCULATIONS

Trial Nos.	I	II	III	Average time in sec
Time taken by water in sec (t_W)				
Time taken by liquid in sec (t_L)				

$$\eta_L = \frac{\eta_w \, t_L d_L}{t_w d_w}$$

Liquid Number =

Room Temperature = …………..ºC

Where t_w = time taken by water =__________________ sec

t_L = time taken by liquid = __________________ sec

d_w = density of water =__________________ g / cc

d_L = density of liquid =__________________ g / cc

η_w = co – efficient of viscosity of water =_________________ milli poise

η_L = co – efficient of viscosity of liquid =_________________ milli poise

$$\eta_L = \frac{\eta_w \, t_L d_L}{t_w d_w} = \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_$$

= ………………milli poise

RESULT:

The viscosity co – efficient of given liquid = _______ milli poise

PROCEDURE:

1. Transfer the known volume (say 15 ml) of the given organic liquid taken in a burette into the viscometer. Suck the organic liquid above the upper mark ‘A’ in the viscometer and allow it to flow freely through a capillary. Start the stop clock when the liquid level just crosses the upper mark ‘A’ and stop it when the liquid level just crosses the lower mark **‘B’** in the viscometer. Note down the time of flow of liquid in seconds (t_l) and repeat the experiment to get agreeing values.
2. Pour out the organic liquid and rinse the viscometer in acetone again. Dry it in an electric oven and fix the viscometer vertically to a stand.
3. Transfer a known volume (say 15 ml) of distilled water taken in a separate burette into a viscometer. Note down the time of flow of water (t_w) in seconds as similar to the liquid and repeat the experiment to get agreeing values.
4. Determine the viscosity co-efficient of the given organic liquid using the relation,

$$\eta_L = \frac{\eta_w \, t_L d_L}{t_w d_w}$$ and the given values of η_w, d_w and d_L.

Note: -Transfer the organic liquid into the bottle after the experiment is completed and rinse the viscometer with acetone and keep it in an electric oven.

OBSERVATIONS AND CALCULATIONS

Volume of Copper sulphate in ml	Concentration of copper sulphate in mg / ml	Concentration of copper in mg / ml	Absorbance (A) **or** Optical density
0	0	0	0.0
5	3.93 x 5 = 19.65	05 x 1 = 05	
10	3.93 x 10 = 39.30	10 x 1 = 10	
15	3.93 x 15 = 58.95	15 x 1 = 15	
20	3.93 x 20 = 78.60	20 x 1 = 20	
25	3.93 x 25 = 98.25	25 x 1 = 25	
Test solution C=	3.93x	x1=	

Note: Molecular weight of copper sulphate = 249.54.

Calculation:

1. **Atomic weight of copper = 63.54**

1.0 ml of stock solution = 3.93 mg of copper sulphate

249.54 gm of $CuSO_4$ = 63.54 g of copper

$$\therefore 3.93 \text{ mg of } CuSO_4 = \frac{63.54 \times 3.93}{249.54} = 1.0 \text{ mg of Copper.}$$

1ml of Stock Solution ($CuSO_4$) = 3.93 mg of $CuSO_4$ = 1.0 mg of Cu

C= concentration of copper in test solution = ___________mg / ml

V ml of copper sulphate = **C** mg of copper

$$\therefore \text{ V ml of copper sulphate } = \mathbf{V} = \frac{C}{1.0} = \frac{\_\_\_\_\_\_\_\_\_\_}{1.0} = \_\_\_\_\_\_\_ \text{ml}$$

Hence, the volume of test solution given = **V** = _____________ ml

EXPERIMENT NO. 3

Date: ☐☐☐☐☐☐☐☐

COLORIMETRIC ESTMATION OF COPPER

THEORY:

When a monochromatic light of intensity I_o is incident on a transparent medium, a part of light is absorbed by the media (I_a**),** a part of light is reflected (I_r) and the remaining part of light is transmitted (I_t).

$\therefore \mathbf{I_o = I_a + I_t + I_r}$ (for a glass – air interface, Ir is negligible)

$\therefore \mathbf{I_o = I_a + I_t}$

The ratio of It / I_o = T called transmittance and log Io / I_t = A which is called absorbance or optical density. The relationship between absorbance (A), concentration (c) (expressed in mol / dm^3) and path length (t) (expressed in cm) is given by Beer – Lambert's law.

$$\mathbf{A = \log I_o / I_t = \varepsilon ct.}$$

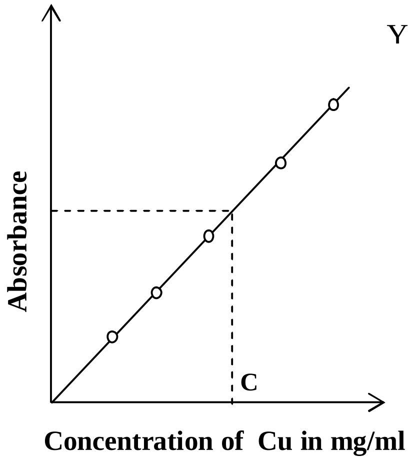
Where **ε** is the molar extension coefficient which is a constant for a given substance at a given wave length and t **=** the path length of the cell tube which is also constant for given cell tube. Therefore, $\mathbf{A \propto c}$**.** Hence plot the graph, absorbance against concentration of the solution gives a straight line.

A series of standard solution of copper salt and test solution is treated with ammonia to get blue cuprammonium complex and is diluted to a definite volume. The absorbance of each of these solution and test solution is measured at **620 nm** since the complex shows maximum absorbance at this wavelength. The absorbance values are plotted against concentration of copper get a calibration curve and concentration of copper in test solution is determined from the graph.

Scale:

X –axis, 1.0 cm = ……….. mg/ml of Cu

Y –axis, 1.0 cm = …………Absorbance



Results:

1. The volume of test solution given = _____________ ml
2. Absorbance of test solution = _____________
3. Concentration of copper in test solution = _____________ mg /ml
4. Concentration of copper sulphate solution = _____________ mg/ml

PROCEDURE:

Transfer the given copper sulphate solution (Stock solution) to a burette and draw out 5,10,15, 20 and 25 ml of the solution into 50 ml standard flasks. Add 5 ml of 1:1 ammonia solution to each of them and also to the test solution given in a separate 50 ml standard flask. Make up the solution to the mark by adding distilled water and mix well for uniform concentration.

Switch on the colorimeter and select the wavelength or λmax = **620nm** and adjust zero absorbance for blank solution .Then measure the absorbance for each standard solution and test solution. A straight line graph is obtained by plotting absorbance against the concentration of copper. From the graph, determine the concentration of copper in the test solution and calculate the volume of copper sulphate in the given test solution.

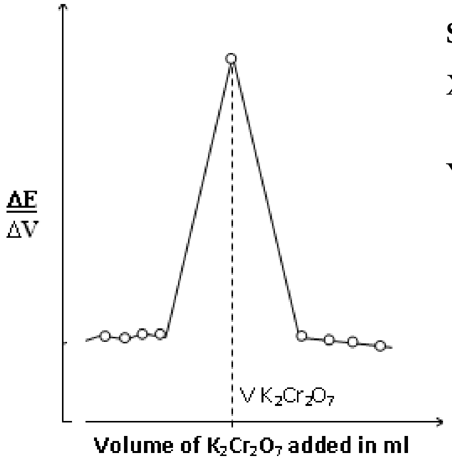
$$CuSO_4 + 4NH3 \longrightarrow [Cu\ (NH_3)_4]\ SO_4 \text{ (blue colour)}$$

(Cuprammonium sulphate complex)

Preparation of blank solution: - Blank solution can be prepared by adding 5.0 ml of 1:1 ammonia and distilled water in 50 ml standard flask. Mix well for uniform concentration.

OBSERVATIONS AND CALCULATIONS

Volume of $K_2Cr_2O_7$ added in ml (V)	E.M.F in mv	ΔE	$\frac{\Delta E}{\Delta V}$	Volume of $K_2Cr_2O_7$ added in ml (V)	E.M.F in mv	ΔE	$\frac{\Delta E}{\Delta V}$
0.0				5.5			
0.5				6.0			
1.0				6.5			
1.5				7.0			
2.0				7.5			
2.5				8.0			
3.0				8.5			
3.5				9.0			
4.0				9.5			
4.5				10.0			
5.0							



Scale :

X –axis, 1.0 cm = _______ ml of $K_2Cr_2O_7$

Y –axis, 1.0 cm = _______ $\frac{\Delta E}{\Delta V}$

$\therefore$ Volume of $K_2Cr_2O_7$ required = V $K_2Cr_2O_7$ = _______ml

EXPERIMENT NO. 4 **Date:** ☐☐☐☐☐☐☐☐

POTENTIOMETRIC ESTIMATION OF MOHR'S SALT (FAS) BY USING STANDARD $K_2Cr_2O_7$ SOLUTION

THEORY:

When the electrode is dipped in the solution, the potential of an electrode depends on the concentration of the ions in the electrolyte. Therefore determination of maximum potential can serve as an indicator in potentiometric analysis. Determination of change of potential with concentration gives more accurate results than volumetric analysis.

When the given electrode is combined with saturated calomel electrode and dipped in the given electrolyte, an electro chemical cell is formed. The emf of this cell is measured using potentiometer. During the titration, the change in emf of the cell or concentration of ions in the electrolyte can be measured. Then $\Delta E / \Delta V$ is plotted against the volume of the titrant, there will be a peak corresponding to the endpoint of the reaction.

$\therefore$ emf of the cell is calculated using Nernst equation,

$$E_{cell} = E^{\circ}_{cell} \frac{+0.0591}{n} \log \frac{[Products]}{[Reactants]}$$

Where E_{cell} = emf of the cell, E°_{cell} standard emf of the cell, n = number of electrons involved in the reaction, [products] = concentration of the products and [reactants] = concentration of the reactants.

Note:

1. Normality of $K_2Cr_2O_7$ = N $K_2Cr_2O_7$ = 0.1 N

2. Equivalent weight of Mohr's salt = 392

$$\therefore \text{ Normality of Mohr's salt solution} = \frac{NK_2Cr_2O_7 \text{ x } VK_2Cr_2O_7}{\text{V Mohr's salt}}$$

$$\text{N Mohr's salt} = \frac{\rule{3cm}{0.4pt}}{25} = \rule{2cm}{0.4pt} \text{ N}$$

Weight per litre of
Mohr's salt crystals = X = N Mohr's Salt x equivalent weight of Mohr's salt

X = ____________ x 392 = ____________ g

Weight per 100 ml of Mohr's salt = X / 10 = ...g

RESULT: Amount of Mohr's salt present in the given solution ....g.

PROCEDURE:

Make up the given Mohr’s salt solution in a 100 ml standard flask to the mark by adding distilled water and mix well for uniform concentration Pipette out 25 ml of Mohr’s salt solution in to a clean 100 ml beaker followed by adding one t.t. of dilute sulphuric acid and stir well using a glass rod.

Connect the platinum and calomel electrodes to the potentiometer, and immerse the electrodes in to the Mohr’s salt solution taken in a beaker. Measure the emf of the solution. Add 0.5 ml of potassium dichromate solution in to the Mohr’s salt solution, stir well and measure the emf of the solution and continue the addition of potassium dichromate added until the sudden raise in the emf of solution occur. Then take few more readings and take the difference in the emf of solution (ΔE) and the volume of potassium dichromate added (ΔV). Plot the graph, ΔE / ΔV against the volume of potassium dichromate added. From the graph, determine the volume of potassium dichromate required to rapid increase in the emf of the solution. Calculate the weight per liter and weight per 100 ml of Mohr’s salt.

$$K_2Cr_2O_7 + 7H_2SO_4 + 6FeSO_4 \longrightarrow 3Fe_2(SO_4)_3 + K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O$$

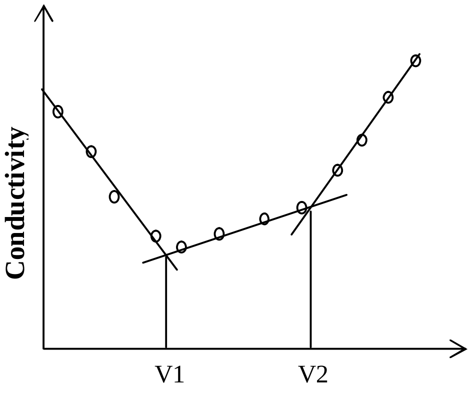
OBSERVATIONS AND CALCULATIONS

Estimation of Acid mixture

Vol. of NaOH in ml	Conductivity $Ohm^{-1}\ cm^{-1}$	Vol. of NaOH in ml	Conductivity $Ohm^{-1}\ cm^{-1}$
0.0		5.5	
0.5		6.0	
1.0		6.5	
1.5		7.0	
2.0		7.5	
2.5		8.0	
3.0		8.5	
3.5		9.0	
4.0		9.5	
4.5		10.0	
5.0			

V_1 =Volume of NaOH required neutralizing HCl =………ml

$(V_2 - V_1)$ =Volume of NaOH required neutralizing CH_3COOH =………ml



Volume of NaOH added in ml

Scale :

X –axis, 1.0 cm = _______ ml of NaOH

Y –axis, 1.0 cm = _______ $ohm^{-1}\ cm^{-1}$

EXPERIMENT NO. 5

Date:

CONDUCTOMETRIC ESTIMATION OF AN ACID MIXTURE USING STANDARD SODIUM HYDROXIDE SOLUTION

THEORY:

Measurement of conductivity can be employed to determine the end point in acid-base titrations. In conductometric titrations there is a sudden change in conductivity of the solution near the endpoint. The end point is determined graphically by plotting conductivity (ordinate) against sodium hydroxide (abscissa).

Conductometric titration may be applied for the determination of acids present in a mixture. In the titration of a mixture of a weak acid (acetic acid) and a strong acid (hydrochloric acid) with a strong base (sodium hydroxide), the conductivity decreases upon adding sodium hydroxide to acid mixture at the initial stages owing to the replacement of highly mobile H^+ ions (mobility: 350 ohm^{-1} m^{-1}) by the less mobile Na^+ ions (mobility: 50$ohm^{-1}m^{-1}$). This trend continues till all the H^+ ions of hydrochloric acid replaced [i.e., the strong acid is neutralized]. Continued addition of sodium hydroxide raises the conductivity moderately, as the weak acid, acetic acid is converted into its salt, sodium acetate. Further addition of sodium hydroxide raises the conductivity steeply due to the presence of free OH^- ions (mobility: 198 ohm^{-1} m^{-1}). The titration curves in the graph given depict the location of the equivalence points.

PROCEDURE:

Make up the given acid mixture in a 100 ml standard flask to the mark by adding distilled water and mix well for uniform concentration. Pipette out 50 ml of the given acid mixture into a clean 100 ml beaker and stir well using a glass rod.

Dip the conductivity cell in the acid mixture and measure the conductivity of the acid mixture .Add 0.5 ml of sodium hydroxide solution from a burette. After each addition, stir the solution gently and note down the conductivity.

Estimation of HCl

Volume of NaOH required to neutralize HCl = V_1 =………….ml

$$\text{Normality of HCl} = \frac{\text{N NaOH x } V_1 \text{ NaOH}}{\text{V Acid Mixture}}$$

$$= \frac{0.2 \text{ x}}{50} = \text{------------N}$$

Weight per liter of HCl = X = N HCl X Equivalent weight of HCl

= --------------------X 36.5

X= ----------------------g

$$\text{Weight per 100 ml of HCl} = \frac{X}{10} = \frac{\text{------------}}{10} = \text{---------- g}$$

Estimation of CH_3COOH

Volume of NaOH required to neutralizing CH_3COOH = ($V_2 - V_1$) =………ml

$$\text{Normality of } CH_3COOH = \frac{\text{N NaOH x } (V_2 - V_1) \text{ NaOH}}{\text{V Acid Mixture}}$$

$$= \frac{0.2 \text{ x}}{50} = \text{------------N}$$

Weight per liter of CH_3COOH = Y = $N_{CH3COOH}$ X Equivalent weight of CH_3COOH

= --------------------X 60

Y= -----------------g

$$\text{Weight per 100 ml of } CH_3COOH = \frac{Y}{10} = \frac{\text{------------}}{10} = \text{---------- g}$$

As the titration proceeds, the conductivity first gradually decreases and then increases slowly and finally increases sharply. Plot the graph conductivity versus volume of sodium hydroxide added. From the graph determined the volume of sodium hydroxide is required to neutralize hydrochloric acid [V_1] and acetic acid [$V_2 - V_1$] in an acid mixture. Calculate the weight per liter and weight per 100 ml of hydrochloric acid and acetic acid respectively.

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

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

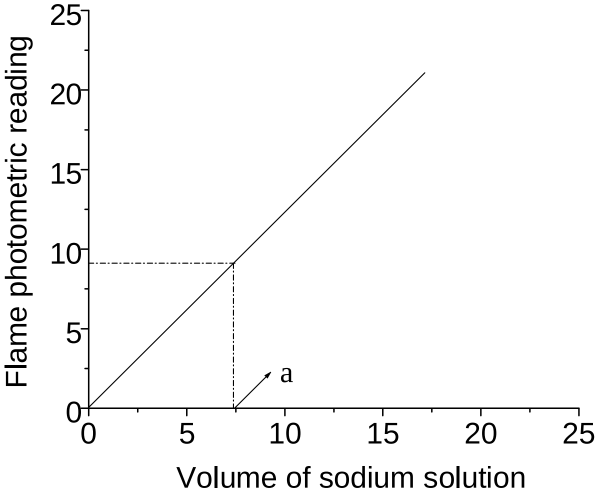
RESULT:

1. Amount of HCl present in the given solution =……………g
2. Amount of CH_3COOH present in the given solution =……………g

OBSERVATION AND CALCULATION

Concentration of the standard solution = 100 ppm

Volume of sodium solution taken	Concentration in ppm	Flame Photometer Reading
5		
10		
15		
20		
Test solution (‘a’ cm^3)		



Unknown volume of NaCl solution =……… (a) cm^3

Therefore, amount of sodium in the test solution $= \frac{a \times 100}{50}$ ppm

$= \frac{\underline{\qquad} \times 100}{50}$ ppm

= ----------- ppm

EXPERIMENT NO. 6 **Date:**

FLAME PHOTOMETRIC ESTIMATION OF SODIUM OR POTASSIUM IN THE GIVEN WATER SAMPLE

THEORY:

Flame photometry is an atomic emission technique used for the detection of alkali and alkali earth metals. If a solution containing metallic salts is sprayed into the flame, electrons in the metal ions of the vapour gain thermal energy and get excited from lower energy level E1 to higher energy levels En, where n = 1, 2, 3 .... From higher energy state these electrons return to the ground state by emitting radiations ($En - E1 = h\gamma$) which are the characteristic of each element.

Flame photometric determination of sodium and other elements such as K, Ca, Li, etc is based on the fact that these elements impart characteristic colors to the Bunsen flame and the brightness of the flame varies with the concentration of these elements present in the sample. Flame photometer correlates the emitted radiation with the concentration of these elements. It is simple and rapid method for the estimation of alkali and alkali earth metals. In a flame photometer a suitable filter for a particular element is placed between the flame and the detector. The sample containing the analyte is aspirated into the flame through atomizer. Radiation from the resulting flame is collected by the lens and allowed to pass through an optical filter, which permits only the characteristic radiation of the element under determination to the photo cell. The output from the photo cell indicates the concentration of the element.

Procedure:

Transfer from the burette 5, 10, 15 & 20 cm^3 of standard sodium solution into different 50 cm^3 volumetric flask. Make up all the solutions using distilled water. Stopper the flasks and shake well to get uniform concentration. To the given test solution add distilled water up to the mark and shake well. Switch on the instrument set the filter for 589nm and adjust the air from the compressor to 10 lbs per sq inch using pressure regulator knob. Put on the burner and adjust the flame to clear blue. Dip the capillary tube to distilled water and set the reading to zero by means of zero control knob. Then feed 100 ppm sodium solution and set the reading to 100 by means of span knob. Repeat the process to confirm the accuracy of the calibration.

Feed the standard sodium solutions one by one including the test solutions and note readings. (Between two standard solutions dip the capillary to distilled water so that reading should become zero). Prepare the calibration graph by taking flame photometric reading against the volume of the solution and estimate the amount of sodium in the given test solution.

Similar procedure is followed for the estimation of potassium from potassium carbonate stock solution using the filter 766nm.

Result:

The amount of sodium present in the given test solution is----------- ppm

VOLUMETRIC METHODS OF CHEMICAL ANALYSIS

PART - B

INSTRUCTIONS TO THE STUDENTS:

- ☞ **Read the procedure before commencement of the experiment and do the experiment according to the procedure.**
- ☞ **Handle the glass wares with care.**
- ☞ **Apparatus used : Burette, Pipette, Standard flask, Conical flask, Beaker, Funnel, Washing bottle for volumetric analysis.**
- ☞ **Wash the all apparatus with tap water and rinse them with distilled water before commencement of the experiment.**
- ☞ **Wash all the apparatus with tap water again after the experiment is completed and return them to the instructor.**

DEFINITIONS

Standard solution :-Solution whose normality is known

Standardization :- Determination of the strength of given solution using standard solution.

Estimation:- Determination of the weight of a given substance present in a known volume of a solution.

Normality :- Number of gm equivalent weight of the substance present in one liter of its solution.

Molarity :- Number of gm molecular weight of the substance present in one liter of its solution.

Indicator:- It is a substance which shows the end point of the reaction by its colour change.

Oxidation:- It is process of loss of electrons by atoms or ions.

Reduction:- It is process of gain of electrons by atoms or ions.

Oxidizing agent:- The substance which gain of electrons. E.g., $K_2Cr_2O_7$, $KMnO_4$ etc.

Reducing agent:- The substance which loss of electrons. E.g., Mohr's salt, $FeCl_2$, hypo etc.

OBSERVATIONS AND CALCULATIONS:

Preparation of standard solution of disodium salt of EDTA

Weight of EDTA salt = ____________g

Molarity of EDTA = $\frac{\text{Weight of EDTA salt}}{\text{Molecular weight of EDTA}} \times 4$

$M_{EDTA} = \frac{\quad \times 4}{372.24} =$ __________ (a) M

Determination of total hardness of water

Burette : Standard EDTA solution

Conical flask: 25 ml of hard water + 5ml of NH_4OH – NH_4Cl buffer

Indicator : 2-3 drops of Eriochrome black – T

End point : Wine red to purple blue colour

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of EDTA run down in mls			

$\therefore$ Volume of EDTA required = V= __________ml

NOTE: Molecular weight of $CaCO_3$ = 100

1000 ml of 1M of EDTA = one g molecular weight of $CaCO_3$ (100)

$\therefore$ V ml of (a) M EDTA = X = $\frac{(a)\times V \times 100}{1000}$ = ----------------------

X = ________g of $CaCO_3$

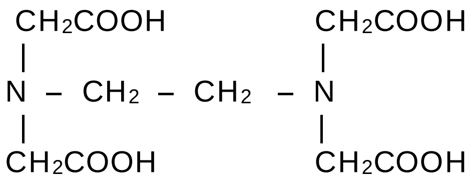
EXPERIMENT NO. 1

Date:

DETERMINATION OF TOTAL HARDNESS OF WATER

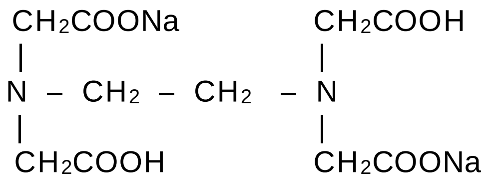
THEORY:

Hardness of water is due to the presence of dissolved salts of calcium and magnesium. The total hardness of water is determined using ethylene diamine tetra acetic acid (EDTA) which form complex with Ca^{2+} and Mg^{2+} ions. The EDTA molecule has two easily replaceable hydrogen atoms and hence the EDTA form complex with metal ion present in water. Reaction can be carried out quantitatively at a pH of 10 using Eriochrome black–T as indicator. Since the reaction involves the liberation of H^+ ions, a buffer mixture has to be used to maintain a pH of 10. The buffer mixture used in the titration is ammonium hydroxide and ammonium chloride. The total hardness of the water is usually expressed in terms of ppm (ie, parts per million) of $CaCO_3$. EDTA is sparingly soluble in water and hence disodium salt of EDTA is used as reagent for preparing the solution. The structures of EDTA (H_4Y) and disodium salt of EDTA (Na_2H_2Y) are given below.



$$\begin{array}{ccccccc} CH_2COOH & & & & & & CH_2COOH \\ | & & & & & & | \\ N & - & CH_2 & - & CH_2 & - & N \\ | & & & & & & | \\ CH_2COOH & & & & & & CH_2COOH \end{array}$$

&



$$\begin{array}{ccccccc} CH_2COONa & & & & & & CH_2COOH \\ | & & & & & & | \\ N & - & CH_2 & - & CH_2 & - & N \\ | & & & & & & | \\ CH_2COOH & & & & & & CH_2COONa \end{array}$$

EDTA(H_4Y)

Disodium salt of EDTA (Na_2H_2Y)

∴ Weight of $CaCO_3$ present
in 25 ml of hard water = X = ________g of $CaCO_3$

∴ Weight of $CaCO_3$ present in one million or
10^6 ml of hard water = Y = $\frac{X \times 10^6}{25}$

Y = $\frac{\qquad \times 10^6}{25}$ = _________ppm

Thus total hardness of water in terms of ppm= Y = _______ppm of $CaCO_3$

Note: Parts per million: ppm :- is the parts of $CaCO_3$ equivalent hardness present per million **or** 10^6 parts of water.

i e: 500 ppm = 500 part of $CaCO_3$ equivalent hardness present per million **or**10^6 parts of water.

PROCEDURE:

I. Preparation of standard solution of disodium salt of EDTA:

Weigh about 1.0 g of disodium salt of EDTA accurately using an electronic balance. Transfer the crystals of EDTA in to a clean 250 ml standard flask using a glass funnel. Dissolve the crystals by adding ½ t.t. of ammonium hydroxide and little distilled water. Make up the solution to the mark by adding distilled water and mix well for uniform concentration. Calculate the molarity of EDTA solution.

II. Determination of total hardness of water:

Pipette out 25ml of the given water sample into a clean conical flask and add 5 ml of buffer solution (ammonium hydroxide and ammonium chloride). Add 2-3 drops of Eriochrome black–T indicator. Titrate this against EDTA solution taken in a burette until the wine red colour changes to purple blue. Note down the volume of EDTA added and repeat the experiment for concordant values. Calculate the total hardness of water in terms of ppm of $CaCO_3$.

REACTIONS:

$M^{2+}(Ca^{2+}$ or $Mg^{2+})$ + In $\longrightarrow$ M – In (wine red colour)
(Hard water) (Indicator) (Metal ion – In complex)

M – In + Na_2H_2Y $\longrightarrow$ M – Y + 2 Na^+ + $2H^+$ + In (blue)
(Metal ion – EDTA complex)

RESULT: The total hardness of the given water sample=……….ppm

OBSERVATIONS AND CALCULATIONS:

I . Preparation of standard solution of disodium salt of EDTA

Weight of EDTA salt = ____________g

$$\text{Molarity of EDTA} = \frac{Weight\ \ of\ \ EDTA\ salt}{Molecular\ weight\ of\ \ EDTA} \times 4$$

$$M_{EDTA} = \frac{\qquad x\ \ 4}{372.24} = \underline{\qquad\qquad} \text{ (a) M}$$

II. Estimation of CaO in cement solution

Burette : Standard EDTA solution

Conical flask : 25 ml of cement solution + 5ml of 1:1 glycerol + 5ml of di-ethylamine + 10 ml of 2N NaOH + shake well

Indicator : 2-3 drops of Pattern - Reeder's indicator

End point : Wine red to purple blue colour

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of EDTA run down in mls			

∴ Volume of EDTA required = V= ________ ml

NOTE: 1. Weight of cement in 250 ml = W = _________g

2. Molecular weight of CaO = 56.08

1000 ml of 1M of EDTA = one gm molecular weight of CaO (56.08)

$$\therefore \text{V ml of (a) M EDTA} = X = \frac{56.08\,x\ \ (a)\ x\ V}{1000} = \frac{56.08\,x\qquad\qquad}{1000}$$

X = _________ g of CaO

EXPERIMENT NO. 2

Date:

DETERMINATION OF CaO IN THE GIVEN SAMPLE OF CEMENT SOLUTION BY RAPID EDTA METHOD

Theory:

Cement contains oxides of calcium, aluminium, magnesium, iron and small amount of silica. Calcium oxide is a prime constituent of cement and the general composition of portland cement is given below.

CaO = 60 – 66%, SiO_2 = 17 – 25%, Al_2O_3 = 3 – 8%, Fe_2O_3 = 2 – 6%, MgO = 0.1- 5.5%, SO_3 = 1 – 3 % and Na_2O & K_2O = 0.5 – 1.5%

In the estimation of calcium in cement, the given cement sample is treated with concentrated hydrochloric acid. The insoluble silica is filtered off and the filtrate which contains calcium ions is titrated against EDTA at a pH of 12 – 14 using Pattern-Reeder's indicator. The latter is used in this titration because Eriochrome black – T indicator forms a very weak complex with calcium ions. Diethylamine is added to maintain a pH of about 12.5 and glycerol is added to get a sharp end point. This method facilitates the determination of calcium only although Mg^{+2} ions are present in the cement solution which can readily form complexes with EDTA (i.e.,Mg^{+2} ions do not react with EDTA in the above conditions)

PROCEDURE:

I.Preparation of standard solution of disodium salt of EDTA:

Weigh about 1.0 g of disodium salt of EDTA accurately using an electronic balance. Transfer the crystals of EDTA into a clean 250ml standard flask using a glass funnel. Dissolve the crystals by adding ½ t.t. of ammonium hydroxide and little distilled water. Make up the solution to the mark by adding distilled water and mix well for uniform concentration. Calculate the molarity of EDTA solution.

$\therefore$ Weight of CaO present in 25 ml of cement solution = X = ________g

$\therefore$ Weight of CaO present in 250 ml of cement solution = Y = $\frac{X \text{ x } 250}{25}$

Y =______________

Y =_______________g

$\therefore$ Percentage of CaO in the cement solution = $\frac{Y \text{ x } 100}{\text{Weight of cement in 250 ml(W)}}$

= ------------------------------

$\therefore$ Percentage of CaO in the cement solution= __________

II. Estimation of calcium oxide:

Pipette out 25ml of the given cement solution into a clean conical flask. Add 5ml of 1:1 glycerol, 5ml of diethyl amine and 10ml of 2N sodium hydroxide. Add 2-3 drops of Pattern- Reeder's indicator. Shake well and titrate this against EDTA solution taken in a burette until wine red colour changes to purple blue. Note down the volume of EDTA added and repeat the experiment for concordant values. Calculate the percentage of calcium oxide in the given cement solution.

REACTIONS:

$$\underbrace{(CaO + SiO_2)}_{\text{Cement}} + 2HCl \longrightarrow CaCl_2 + SiO_2\downarrow + H_2O$$

$$Ca^{2+} + In \longrightarrow \text{Ca – In complex (wine red colour)}$$

$$Ca - In + EDTA \longrightarrow \text{Ca – EDTA complex} + \text{In (blue colour)}$$

RESULT: The Percentage of CaO present in the given cement

solution = __________

OBSERVATIONS AND CALCULATIONS:

I. Preparation of Brass solution:

Weight of brass = W = _________g

II. Estimation of copper

Burette : Standard hypo solution

Conical flask : 25 ml brass solution + NH_4OH drop wise till a bluish white ppt. is formed. Dissolve the ppt. by adding dilute acetic acid + ¼th t.t. of acetic acid in excess + One t.t. of 10% KI + shake well.

Indicator : 2 ml of freshly prepared starch.

End point : Disappearance of blue colour

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of hypo run down in mls			

∴ Volume of hypo required = V= ________ ml

NOTE: **1. Normality of hypo = 0.03N**

2. One gm equivalent weight of copper = 63.54

∴ 1000 ml of 1 N of hypo = one gm equivalent weight of copper (63.54)

∴ V ml of 0.05 N hypo = X = $\dfrac{V \text{ x } 0.03 \text{ N x } 63.54}{1000}$

$$= \frac{0.03 \times 63.54 \times }{1000}$$

X = ___________ g of copper

∴ Weight of copper present in 25 ml of brass solution = X = __________g

EXPERIMENT NO. 3

Date: ☐☐☐☐☐☐☐☐

DETERMINATION OF PERCENTAGE OF COPPER IN BRASS USING STANDARD SODIUM THIOSULPHATE SOLUTION

THEORY:

The chief constituents of brass alloy are copper and zinc. It also contains small quantities of tin, lead and iron. The percentage composition of a typical brass alloy is given below.

Cu = 50 – 90 % , Zn = 20 – 40 % , Sn = 0 - 6% , Pb = 0 - 2% and Fe = 0 – 1% .

A solution of brass is made by dissolving the sample in minimum amount of nitric acid. Excess of nitric acid destroyed by boiling with urea. The solution is neutralised and made in to slight acidic medium. Potassium iodide solution is added and the cupric ion present in the brass solution oxidise potassium iodide to iodine. The iodine liberated is titrated against sodium thiosulphate using starch as indicator. The volume of sodium thiosulphate consumed is measure of the amount of copper present in the solution and sodium thiosulphate is commonly known as hypo.

PROCEDURE:

I. Preparation of Brass solution:

Weigh about 1.2 g of the brass pieces accurately using an electronic balance and transfer it into a clean conical flask. Add two t.t. of 1:1 nitric acid and cover the mouth of the flask with a glass funnel. Warm gently to dissolve the brass piece completely and add two t.t of distilled water. Add about two g of urea and continue the heating until the reddish brown fumes of oxides of nitrogen are expelled completely. Cool the solution to room temperature and transfer the solution of brass in to a clean 250 ml standard flask. Make up the solution to the mark by adding distilled water and mix well for uniform concentration.

∴ Weight of copper present in 250 ml of brass solution = Y = $\frac{X \quad x \quad 250}{25}$

$$Y = \frac{\quad x \quad 250}{25}$$

Y =__________ g

∴ Percentage of copper in the given brass sample = $\frac{Y\ X\ 100}{Weight\ of\ brass(W)}$

= ___________________

= ________

II Estimation of copper:

Pipette out 25 ml of brass solution into a clean conical flask and add dilute ammonium hydroxide drop by drop until bluish white precipitate is formed. Dissolve the ppt. by adding dilute acetic acid drop by drop and then add 1/ 4 t.t. of acetic acid in excess. Add one t.t. of 10% potassium iodide solution and shake well. Titrate the liberated iodine against standard sodium thiosulphate solution taken in a burette using starch as indicator towards the end point. (Add starch when brown solution changes to whitish yellow). Disappearance of blue colour is the end point. Note down the volume of hypo added and repeat the experiment for concordant values. Calculate the percentage of copper in the given sample of brass.

REACTIONS :

$$Cu + 4\,HNO_3 \xrightarrow{\text{heat}} Cu(NO_3)_2 + 2\,H_2O + 2\,\underset{\text{(Reddish brown)}}{NO_2\uparrow}$$

$$2\,Cu(NO_3)_2 + 4\,KI \longrightarrow \underset{\text{(Cuprous iodide)}}{Cu_2I_2\downarrow} + 4\,KNO_3 + I_2\uparrow \text{ (Brown)}$$

$$\underset{\text{(Hypo)}}{2\,Na_2S_2O_3} + I_2 \longrightarrow 2\,NaI + Na_2S_4O_6 \text{ (sodium tetra thionate)}$$

$$I_2 + \text{starch} \longrightarrow \text{Starch-iodide complex (Deep blue)}$$

RESULT: The Percentage of copper present in the given brass sample=………

OBSERVATIONS AND CALCULATIONS:

I. Preparation of standard potassium dichromate solution :

Weight of $K_2Cr_2O_7$ crystals = ____________g

$$\text{Normality of } K_2Cr_2O_7 = \frac{\text{Weight of } K_2Cr_2O_7 \times 4}{\text{Equivalent weight of } K_2Cr_2O_7}$$

$$N\ K_2Cr_2O_7 = \frac{\qquad\qquad \text{x } 4}{49} = \underline{\qquad\qquad}\text{(a) N}$$

II. Estimation of iron

Burette : Standard $K_2Cr_2O_7$ solution

Conical flask : 25 ml of haematite ore solution + ¼ t.t concentrated HCl + heat + $SnCl_2$ drop wise till yellow colour changes to colourless + 5.6 drops of $SnCl_2$ in excess + cool the solution to room temperature + 10 ml of saturated $HgCl_2$ + shake well + one t.t. of distilled H_2O

Indicator : Potassium ferricyanide as external indicator

End point : Failure to observe blue colour when a drop of reacting mixture is brought in contact with the indicator placed on paraffined paper

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of $K_2Cr_2O_7$ run down in mls			

∴ Volume of $K_2Cr_2O_7$ required = V = _________ ml

NOTE: **1. Weight of haematite ore in 250ml = W = __________g**

2. Equivalent weight of iron = 55.85

EXPERIMENT NO. 4

Date:

DETERMINATION OF IRON IN A GIVEN SAMPLE OF HAEMATITE ORE SOLUTION USING $K_2CR_2O_7$ CRYSTALS BY EXTERNAL INDICATOR METHOD

THEORY:

Haematite is an important ore of iron containing mainly, ferric oxide (Fe_2O_3) and small amount of silica (SiO_2). A known weight of the ore is digested with hydrochloric acid and the insoluble residue mainly containing silica is removed by filtration. The filtrate is diluted to 250 ml and is supplied for the estimation. Iron is present in the solution as Fe^{3+} (ferric) ions ant it is reduced to Fe^{2+} (ferrous) ions using stannous chloride ($SnCl_2$) in hot condition in presence of concentrated HCl. The excess of stannous chloride is oxidized by adding with mercuric chloride solution. The ferrous ions in the resulting solution are titrated against standard potassium dichromate using potassium ferricyanide as external indicator. Potassium ferricyanide produces an intense deep blue colour with ferrous ions due to the formation of ferriferrocyanide complex.

PROCEDURE:

a) Preparation of standard solution of potassium dichromate:

Weigh about 1.2 g of potassium dichromate crystals accurately using an electronic balance. Transfer the crystals of potassium dichromate into a clean 250ml standard flask using a glass funnel. Dissolve the crystals by adding distilled water and make up the solution to the mark by adding distilled water. Mix well for uniform concentration and calculate the normality of potassium dichromate solution.

1000 ml of 1 N of $K_2Cr_2O_7$ = one gm equivalent weight of iron (55.85)

$\therefore$ V ml of (a) N $K_2Cr_2O_7$ = X = $\dfrac{55.85 \text{ x } (a) \text{ x } V}{1000}$

$$X = \frac{55.85 \times \qquad\qquad}{1000} = \underline{\qquad\qquad} \text{ g of iron}$$

$\therefore$ Weight of iron present in 25 ml of haematite solution = X = ________g

$\therefore$ Weight of iron present in 250 ml of

haematite solution = Y = $\dfrac{X \text{ x } 250}{25}$ = ________

Y = _________g

$\therefore$ Percentage of iron in the given

haematite solution = $\dfrac{Y \times 100}{Weight\ of\ haematite\ ore\ in\ 250\ ml\ (W)}$

= ______________________

= _____________

b) Estimation of iron:

Pipette out 25ml of the haematite ore solution into a clean conical flask. Add ¼ t.t of concentrated hydrochloric acid and heat the solution nearly to boiling. Add stannous chloride drop by drop taken in a separate burette till yellow colour solution becomes colourless and then add 6-8 drops of stannous chloride in excess. Cool the solution to lab temperature and add 10ml of saturated mercuric chloride at once. Shake well and a silky white precipitate is formed. (If no ppt or black ppt is formed, reject and repeat the experiment). Add one t.t. of distilled water and titrate this against standard potassium dichromate solution taken in a burette, using potassium ferricyanide as external indicator. The end point is failure to observe blue colour when one drop of the titrated mixture is brought in contact with a drop of the indicator placed on paraffin paper. Note down the volume of potassium dichromate added and repeat the experiment for concordant values. Calculate the percentage of iron in the given sample of haematite ore solution.

REACTIONS:

$$\underset{\text{(Haematite ore)}}{Fe_2O_3} + 6HCl \xrightarrow{\text{heat}} \underset{\text{[Ferric chloride)}}{2FeCl_3} + 3\,H_2O$$

$$FeCl_3 + SnCl_2 \longrightarrow 2FeCl_2 + SnCl_4$$

$$SnCl_2 + 2\,HgCl_2 \longrightarrow Hg_2Cl_2 + SnCl_4$$

$$K_2Cr_2O_7 + 14HCl + \underset{\text{(Ferrous chloride)}}{6FeCl_2} \longrightarrow 6\,FeCl_3 + 2KCl + 2CrCl_3 + 7H_2O$$

$$3FeCl_2 + \underset{\text{(Potassium Ferricyanide)}}{2\,K_3\,[Fe(CN)_6]} \longrightarrow 6KCl + \underset{\text{(Ferri Ferrocyanide complex)}}{Fe_3\,[Fe\,(CN)_6]_2} \text{ (deep blue)}$$

RESULT: The Percentage of iron present in the given haematite ore solution =…….

OBSERVATIONS AND CALCULATIONS:

I. Preparation of Mohr's salt or ferrous ammonium sulphate (FAS) solution:

Weight of FAS crystals =__________g

$$\textbf{Normality of FAS} = \frac{\text{Weight of FAS} \times 4}{\text{Equivalent weight of FAS}}$$

$$\text{N FAS} = \frac{\quad x\ 4}{392} = \_\_\_\_\_\_\_\_\_(a)\ \text{N}$$

II. Estimation of COD or back titration

Burette : Standard FAS solution

Conical flask : 25 ml of waste water +25 ml of standard $K_2Cr_2O_7$ solution + 10 ml of 1:1 H_2SO_4 [$(Ag)_2SO_4$ + $HgSO_4$]

Indicator : 2 – 4 drops of ferroin indicator

End point : Blue green to reddish brown colour

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of FAS run down in mls			

∴ Volume of FAS solution required for back titration = V_1 = _____ ml

EXPERIMENT NO. 5 **Date:**

DETERMINATION OF CHEMICAL OXYGEN DEMAND (COD) OF THE GIVEN INDUSTRIAL WASTE WATER SAMPLE

THEORY:

The chemical oxygen demand (COD) test is extensively employed for the measurement of the pollution strength of industrial wastes. Chemical oxygen demand is a measure of the total quantity of oxygen required for oxidation of organic compounds of wastes to CO_2 and water by a strong oxidising agent. This parameter is particularly valuable in surveys designed to determine the control losses to sewer systems. Results may be obtained within a relatively short time and measures taken to correct error on the day they occur.

Waste water contains organic impurities which include straight chain aliphatic and aromatic compounds, such as alcohols, acids, amines, pyridine and other oxidisable materials. They are oxidise more effectively when silver sulphate is added as a catalyst. But silver sulphate reacts with chlorides in the waste water to form precipitates which are oxidised partially by this procedure. This difficulty is overcome by adding mercuric sulphate to the sample.

PROCEDURE:

I. PREPARATION OF STANDARD MOHR'S SALT OR FAS SOLUTION:

Weigh about 1.0 g of Mohr's salt crystals accurately using an electronic balance and transfer the crystals into a clean 250ml standard flask using a glass funnel. Dissolve the crystals by adding ½ t.t. of dilute sulphuric acid and little distilled water. Make up the solution to the mark by adding distilled water. Mix well for uniform concentration and calculate the normality of Mohr's salt solution.

IV Blank titration or (FAS > < $K_2Cr_2O_7$)

Burette : Standard FAS solution

Conical flask : 25 ml of $K_2Cr_2O_7$ solution + 10 ml of 1:1 H_2SO_4

Indicator : 2 – 4 drops of ferrion indicator

End Point : Blue green to reddish brown colour

∴Volume of FAS solution
required for blank titration = V_2 = Final B.R - Initial B.R

V_2 = _______=_______ml

∴Volume of $K_2Cr_2O_7$ required for the oxidation
of organic compounds of waste water = $V_2 - V_1$ = ________ = _____ ml

1000 ml of 1 N of FAS = 1000 ml of 1N $K_2Cr_2O_7$ solution = 8 gms of oxygen

$$V_2 - V_1 \text{ ml of (a) N FAS} = X = \frac{8\times (a) \times V_2 - V_1}{1000} = \frac{8 \text{ x } \underline{\qquad\qquad}}{1000}$$

X = _________g of oxygen

∴ Amount of O_2 required to oxidise organic
compounds present in 25 ml of waste water = X = ______g

∴ Amount of O_2 required to oxidize organic
compounds present in 1000ml of waste water = Y = $\frac{X \times 1000}{25}$

= ________

Y = _______g

∴ COD of waste water = Y x 1000 = ________ x 1000

∴ COD of waste water = _____ mg of oxygen

II Determination of COD of waste water or back titration:

Pipette out 25 ml of the industrial wastewater and 25ml of potassium dichromate solution into a clean conical flask. Add 10 ml of 1:1 sulphuric acid containing silver sulphate and mercuric sulphate. Add 2 -4 drops of ferroin indicator and titrate against standard Mohr's salt solution taken in a burette until blue-green colour changes to reddish-brown. Note down the volume of Mohr's salt solution added for unreacted potassium dichromate solution (V_1) and repeat the experiment to get concordant values. Using blank titre value determines the volume of potassium dichromate required to oxidise organic compounds and calculate the COD of the given industrial waste water.

III. Blank titration or Mohr's salt > < $K_2Cr_2O_7$:

Pipette out 25ml of standard potassium dichromate solution into a clean conical flask and add 10ml of 1:1 sulphuric acid. Add 2 – 4 drops of ferrion indicator and titrate this against Mohr's salt solution taken in a burette until blue–green colour changes to reddish brown. Note down the volume of Mohr's salt solution required for blank titration (V_2).

REACTIONS:

$$\underset{\text{(organic compounds)}}{\text{Industrial wastewater}} + \underset{(K_2Cr_2O_7)}{\text{oxidising agent}} \xrightarrow[H_2SO_4]{\text{heat}} H_2O + CO_2\uparrow$$

$$\underset{\text{(Excess)}}{K_2Cr_2O_7} + 7H_2SO_4 + 6FeSO_4 \longrightarrow 3Fe_2(SO_4)_3 + K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O$$

$$\underset{\text{(Blue green)}}{\text{Ferric ion indicator complex } (Fe^{3+})} \underset{K_2Cr_2O_7}{\overset{FAS}{\rightleftharpoons}} \underset{\text{(Reddish brown)}}{\text{ferroin indicator } (Fe^{2+})}$$

RESULT: The COD value of the given waste water= _____ mg of oxygen

OBSERVATIONS AND CALCULATIONS:

IV. Preparation of bleaching powder solution:

Weight of bleaching powder taken = W = _________g

V. Estimation of chlorine:

Burette : Standard hypo solution
Conical flask : 25 ml bleaching powder solution + One t.t. of 2% KI + 5ml of glacial acetic acid, shake well.
Indicator : 2 ml of freshly prepared starch.
End point : Disappearance of blue colour

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of hypo run down in ml			

∴ Volume of hypo required = V= ________ ml

NOTE: **1. Normality of hypo = 0.05 N**
2. One g equivalent weight of chlorine = 35.46

∴ 1000 ml of 1 N of hypo = one gm equivalent weight of chlorine (35.46)

∴ V ml of 0.05 N hypo = X $= \dfrac{V\ X\ 0.05\ N\ X\ 35.46}{1000}$

$$= \frac{0.05\ \ X\ 35.46\ X}{1000}$$

X = ___________ g of chlorine

∴ Weight of chlorine present in 25 ml of bleaching powder solution = X = __________g

EXPERIMENT NO. 6

Date: ☐☐☐☐☐☐☐☐

ESTIMATION OF PERCENTAGE OF CHLORINE IN BLEACHING POWDER USING STANDARD SODIUM THIOSULPHATE SOLUTION (Iodometric method)

THEORY:

Bleaching powder is mainly, a mixture of calcium hypo chlorite [$Ca(OCl)_2$] and the basic chloride [$CaCl_2$, $Ca(OH)_2.H_2O$]. The active part of the bleaching powder is hypochlorite from which the chlorine is liberated for bleaching powder. The available chlorine means the chlorine liberated by the reaction of dilute acids and is expressed as the percentage by weight of the bleaching powder.The available chlorine can be estimated by iodometric method. The bleaching powder solution of suspension is treated with an excess of potassium iodide and strongly acidified with acetic acid.

$$Ca(OCl_2) + 2CH_3COOH \longrightarrow Ca(CH_3COO)_2 + H_2O + Cl_2$$

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

The liberated iodine is treated with standard solution of hypo

$$2Na_2S_2O_3 + I_2 \longrightarrow NaI + Na_2S_4O_6$$

$$I_2 + \text{Starch} \longrightarrow \text{Starch - Iodide complex (Deep blue)}$$

PROCEDURE:

I. PREPARATION OF STANDARD BLEACHING POWDER SOLUTION:

Weigh about 2 g of the bleaching powder accurately using an electronic balance and transfer it into a clean mortar. Add a little water and rub the mixture into a smooth paste. Add a little more water and mix with the pestle, allow the mixture to settle and transfer the milky liquid into 250 ml standard flask. Grind the residue with a little more water and repeat the operation until complete sample has been transferred to the flask in a state of very fine suspension. Makeup to the mark with distilled water and mix well for uniform concentration.

∴ Weight of chlorine present in 250 ml of bleaching powder solution =

$$Y = \frac{X \quad x \quad 250}{25}$$

$$Y = \frac{\quad x \quad 250}{25}$$

Y =__________ g

∴ Percentage of chlorine in the given bleaching powder sample

$$= \frac{Y\ X\ 100}{Weight\ of\ bleaching\ powder(W)}$$

= __________________

= ________

II ESTIMATION OF CHLORINE:

Pipette out 25 ml of bleaching powder solution into a clean conical flask and add 10ml of 2% KI solution. Add 5ml of glacial acetic acid and titrate the liberated iodine against standard sodium thiosulphate solution taken in a burette using starch as indicator towards the end point. Disappearance of blue colour is the end point. Note down the volume of hypo added and repeat the experiment for concordant values. Calculate the percentage of chlorine in the given bleaching powder.

Result: Percentage of chlorine in the given bleaching powder is ……….

VIVA QUESTIONS

Part-A

1. What is weak acid ?
2. What is pKa of a weak acid ?
3. Define pH.
4. What is Ka?
5. How are pH and pKa related?
6. What are the electrodes used in the measurement of pH for the determination of pKa?
7. Why half equivalent point is taking for calculating pKa value of weak acid?
8. What is viscosity?
9. What is viscosity coefficient of a liquid?
10. What is the SI unit of viscosity coefficient?
11. How does the viscosity vary with temperature?
12. Write the expression used to calculate the viscosity coefficient of liquid.
13. Why is the same volume of water and liquid used in the determination of viscosity? coefficient of liquid?
14. Which reference electrode is used in the determination of ferrous ammonium sulphate potent metrically?
15. What is meant by transmittance?
16. What is absorbance?
17. Define Bear – Lambert’s law.
18. What is the relation between absorbance and concentration?
19. What is a blank solution?
20. Which compound of copper is responsible for the deep blue colour in the colorimetric determination of copper?
21. Why is ammonia added to copper sulphate solution in the colorimetric determination of copper?
22. Why is the estimation of copper done at 620 nm wavelengths?
23. What is the wave length used in the colorimetric determination of iron?
24. Why is potassium thiocyanate solution added to ferric alum solution in the colorimetric determination of iron?
25. Which compound of iron is responsible for the red colour in the colorimetric determination of iron?
26. What is conductance?
27. What is the unit for conductance?
28. What is emf of the cell?
29. What is cell constant? Mention its unit.
30. Why is conductance of HCl decreases on adding NaOH before the end point?
31. Why is the conductance of HCl increases on adding NaOH after the end point?
32. What is an electrolyte?
33. What are types of electrolyte? And give example for each?

Part-B

1. What is an acid and base?
2. What is meant by titration?
3. What is end point?
4. What is a standard solution?
5. What is standardization?
6. What is estimation?
7. Define normality.
8. Define molarity.
10. What is an indicator? And mention the different types of indicators.
11. What is an oxidation and reduction?
12. What is an oxidising and reducing agents?
13. What is an hard water?
14. How is hardness of water caused?
15. What are the types of hard water?

16. What are the causes of temporary and permanent hardness of water?
17. How is temporary and permanent hardness of water removed?
18. How do you express the total hardness of water?
19. Name the metal ion indicator.
20. What is EDTA?
21. What are buffer solutions?
22. Mention types of buffer solutions.
23. Whyisammonia-sodiumhloridebuffer solution is added in the determination of total hardness of water?
24. What is the end point in the determination of total hardness of water?
25. What are the constituents of cement?
26. Which indicator used in the determination of CaO in cement solution?
27. Why the diethylamine and glycerol added to the cement solution?
28. What is an ore?
29. What is the main constituent of haematite ore?
30. What is the role of stannous chloride in the determination of iron in haematite ore solution?
31. Why is mercuric chloride added in the determination of iron in haematite ore solution?
32. What happens when the excess of stannous chloride is not removed in the determination iron in haematite ore solution?
33. What is the indicator used in the determination of iron in haematite ore solution?
34. Whyis potassium ferricyanide cannot be used as an internal indicator in the analysis of haematite ore?
35. What is the colour of $K_3[Fe(CN_6]$ with ferrous ion?
36. Why is the colour of the indicator drop remains the same at the end point in the determination of iron in haematite ore?
37. What is an alloy?
38. What are the constituents of brass?
39. How is a brass solution prepared?
40. What is the purpose of adding urea to the brass solution?
41. Why is ammonium hydroxide added to the brass solution in the determination of copper?
42. Why is acetic acid added to the brass solution in the determination of copper?
43. Why is KI added to the brass solution although copper is present in brass solution?
44. Why is the blue solution of brass turns brown upon the addition of KI solution?
45. Why is starch indicator added towards the endpoint in iodometric titrations?
46. What is the white precipitate left at the end point in the determination of copper in brass?
47. What is hypo?
48. What is Mohr’s salt?
49. What are the salts present in Mohr’s salt?
50. Why is sulphuric acid added during the preparation of Mohr’s salt solution?
51. What is a hydrolysis?
52. What is chemical oxygen demand (COD)?
53. What is biological oxygen demand (BOD)?
54. What is the role of silver sulphate in the determination of COD?
55. What is role mercuric sulphate in the determination COD?
56. What is the indicator used in COD experiment?
57. What is the colour change at the end point in the determination of COD?
58. Why COD value always greater then BOD value.
59. What is the chemical name of bleaching powder?
60. How to prepare bleaching powder?
61. Which ions are present in bleaching powder solution?
62. How to estimate available chlorine in bleaching powder?
63. What happens when bleaching powder reacts with water?
64. Why is starch used in the estimation of chlorine in bleaching powder?
65. Give any two commercial uses of bleaching powder.

SCHEME OF VALUATION

DESCRIPTION	Part-A	Part-B	Max. marks
Procedure write up	08	07	15
Conduction	26	26	52
Calculation, graph works and Result	09	09	18
Viva- Voice	07	08	15

Part A					
Potentiometry, Colorimetry and Flame Photometry		P^{ka} and Viscosity		Conductometry	
Error (cm^3)	Marks	Error (cm^3)	Marks	Error (cm^3)	Marks
±0.5	13 + 13	±5	13 + 13	±0.5	13 + 13
±0.5 to 0.6	11 + 11	±5.1 to 6	11 + 11	±0.51 to 0.6	11 + 11
±0.6 to 0.7	9 + 9	±6.1 to 7	9 + 9	±0.61 to 0.7	9 + 9
±0.7 to 0.8	7 + 7	±7.1 to 8	7 + 7	±0.71 to 0.8	7 + 7
±0.8 to 1.0	5 + 5	±8.1 to 10	5 + 5	±0.81 to 1.0	5 + 5
> ± 1.0	Zero	> ± 10	Zero	> ±1.0	Zero
Graph:5 Calculation: 3		Pka: Two Graphs: 5+3 Viscosity: 8		Graph: 4 Calculation: 4	

Part B	
Total hardness, CaO in cement, Cu in Brass, Fe in Haematite and COD	
Error (cm^3)	Marks
±0.2	13 + 13
±0.3	11 + 11
±0.4	9 + 9
±0.5	7 + 7
± 0.6	5 + 5
> 0.6	Zero
Calculation: 08 Note: Best TWO TITRE values should be considered for valuation	

Overwritten values are not considered for valuation.