"Jai Sri Gurudev"
Sri Adhichunchanagiri Shikshana Trust ®
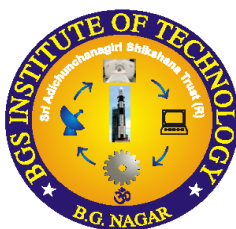
B.G.S.INSTITUTE OF TECHNOLOGY

B.G NAGARA - 5 71448
MANDYA

ENGINEERING CHEMSITRY
LABORATORY MANUAL

I/II SEMESTER B.E.

PREPARED BY :
FACULTY, DEPARTMENT OF CHEMISTRY

Engineering Chemistry Laboratory

Sub Code: 18CHEL16/18CHEL 26   Time : 03 Hours
Marks : 80

(For Examination, any one experiment from Part- A and a common experiment from Part - B shall be set)

PART – A

PART – B

References Books:

1. Laboratory manual in Engineering Chemistry Sudharani, Dhanpatrai Publishing Company.
2. Vogel's Text Book of Quantitative Chemical Analysis revised by G.H.Jeffery, J. Bassett, J. Mendham and R.C Denney.

3. **INSTRUCTIONS FOR INSTRUMENATAL METHOD OF ANALYSIS**

❖ Read the procedure before commencement of experiment and do the experiment according to the procedure.

❖ Instruments used for Part – A experiments are:

❖ pH meter, Potentiometer, Colorimeter, Conductivity meter and Viscometers.

❖ Wash the micro burette, pipette and beaker with tap water and rinse with distilled water before commencement of the experiments.

❖ Wash the electrodes or cells with distilled water and wipe them with tissue paper before commencement of the experiments.

❖ Switch off the instruments after experiment is completed.

❖ Wash the electrodes or cells with distilled water and dip them in distilled water taken in a 100 cm^3 beaker after the experiments is completed.

❖ Wash the micro burette, pipette, standard flask and beaker with tap water again after the experiment is completed and return them to the instructor.

INSTRUCTIONS FOR VOLUMETRIC METHOD OF ANALYSIS

❖ 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.

❖ 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 after the experiment is completed and return them to the instructor

PART-A

INSTRUMENATAL

METHOD OF ANALYSIS

Volume of $K_2Cr_2O_7$	E.M.F (E) in mV	ΔE	ΔV	$\Delta E/\Delta V$
0.0		**		
0.5				
1.0				
1.5				
2.0				
2.5				
3.0				
3.5				
4.0				
4.5			0.5	
5.0				
5.5				
6.0				
6.5				
7.0				
7.5				
8.0				
8.5				
9.0				
9.5				
10.0				

INSTRUMENTAL METHODS OF CHEMICAL ANALYSIS

PART-A

EXPERIMENT NO. 1   **Date:**

POTENTIOMETRIC ESTIMATION OF FAS 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.

The given electrode is combined with saturated calomel electrode and dipped in the 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 end point of the reaction.

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

$$E_{cell} = E°_{cell} \frac{+\ 0.0591}{n} \log \frac{[Pr\,oducts]}{[Re\,ac\tan ts]}$$

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.
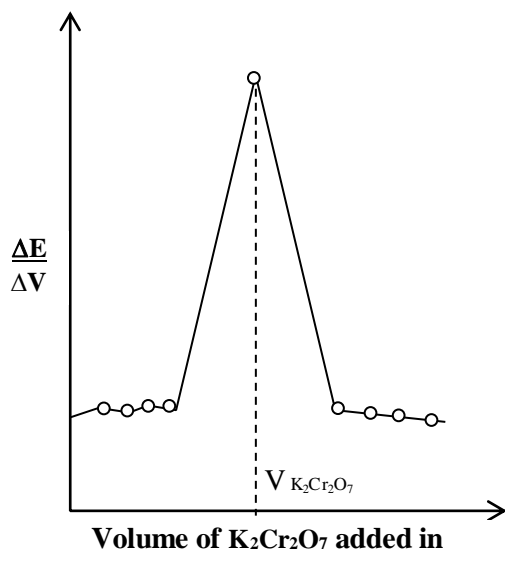
Scale :

X –axis, 1.0 cm = _____ cm^3

Y –axis, 1.0 cm = _____ units

Volume of $K_2Cr_2O_7$ added in

∴Volume of $K_2Cr_2O_7$ required = $V_{K2Cr2O7}$ = _____ cm^3

 Note:

1. Normality of $K_2Cr_2O_7$ = $N_{K_2Cr_2O_7}$ = 0.1 N

2. Equivalent weight of Mohr's salt = 392

∴ Normality of Mohr's salt solution = $\dfrac{NK_2Cr_2O_7 \ x \ VK_2Cr_2O_7}{V \ Mohr's \ salt}$

$N_{Mohr's \ salt}$ = $\dfrac{\rule{4cm}{0.4pt}}{25}$ = _____ N

Weight per liter of
Mohr's salt crystals = X = $N_{Mohr's \ Salt}$ x equivalent weight of Mohr's salt

X = _____ x 392 = _____ g

∴Amount of Mohr's salt present in the given solution is = …………………g.

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

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PROCEDURE:

Transfer the given amount of Mohr's salt solution (25 cm^3) in to a clean 100 cm^3 beaker. to that add one test tube of dilute sulphuric acid.

Switch on the potentiometer and adjust the temperature knob to the lab temperature. Connect the platinum and calomel electrodes to the potentiometer. Wash the electrodes with distilled water and immerse the electrodes in to the Mohr's salt solution taken in a beaker. Measure the emf of the solution in terms of **mV**. Add 0.5 cm^3 of potassium dichromate solution at a time taken in a micro burette into the Mohr's salt solution. Stir the solution and measure the emf of the solution and continue the addition potassium dichromate added until the sudden raise in the emf. 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, $\Delta E/\Delta V$ 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 amount of Mohr's salt given in the solution.

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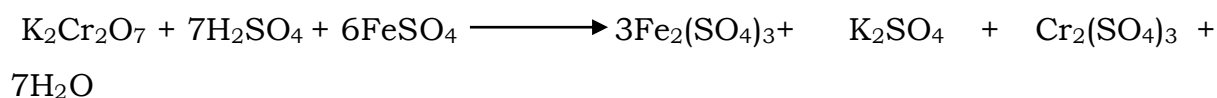
(FAS)

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

OBSERVATIONS AND CALCULATIONS

| Volume of copper sulphate in cm³	Concentration of copper sulphate in mg / cm³	Concentration of copper in mg / cm³	Absorbance (A) or OD at 620nm
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			

Note: Molecular weight of copper sulphate pentahydrate = 249.54.

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

1.0 cm³ of stock solution = 3.93 mg of copper sulphate

249.54 g of $CuSO_4$ $5H_2O$ = 63.54 g of copper

∴ 3.93 mg of $CuSO_4$ = $\dfrac{63.54 \times 3.93}{249.54}$ = 1.0 mg of Copper.
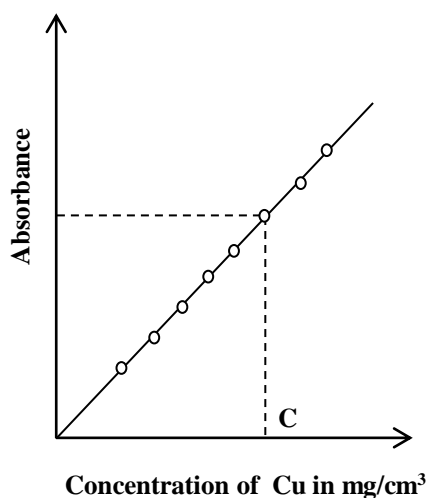
Scale :

X –axis, 1.0 cm = _____ mg/cm³

Y –axis, 1.0 cm = …………… OD

EXPERIMENT NO. 2   **Date:**

DETERMINATION OF COPPER BY COLORIMETRIC METHOD

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).

$$\therefore \textbf{I}_\textbf{o} = \textbf{I}_\textbf{a} + \textbf{I}_\textbf{t} + \textbf{I}_\textbf{r} \quad \textbf{(}\text{for a glass – air interface, Ir is negligible)}$$

$$\therefore \textbf{I}_\textbf{o} = \textbf{I}_\textbf{a} + \textbf{I}_\textbf{t}$$

The ratio of I_t/I_o = T called transmittance and log I_o/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.

$$\textbf{A} = \textbf{log I}_\textbf{o} \textbf{ / I}_\textbf{t} = \boldsymbol{\varepsilon} \textbf{ ct.}$$

Where $\boldsymbol{\varepsilon}$ is the molar extension coefficient which is a constant for a given substance at a given wave length and t is the path length of the cell tube which is also constant for given cell tube. Therefore, **A α 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.

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∴ **1.0 cm³ of CuSO₄ =   3.93 mg of CuSO₄ = 1.0 mg of copper**

C= concentration of copper in test solution   = _____mg / cm³

V cm³ of copper sulphate   = C mg of copper

∴   V cm³ of copper sulphate = V = $\dfrac{C}{1.0}$ = $\dfrac{\rule{2cm}{0.4pt}}{1.0}$ =
_____cm³

Hence, the volume of test solution given   =   V   =   _____ cm³

PROCEDURE:

Transfer the given copper sulphate solution (Stock solution) to a burette and draw out 5, 10, 15, 20 and 25 cm^3 of the solution into 50 cm^3 standard flasks. Add 5 cm^3 of 1:1 ammonia solution to each of them and also to the *test solution given in a separate 50 cm³ standard flask*. Make up the solution to the mark by adding distilled water and mix well for uniform concentration. *Prepare a blank solution by adding 5 cm³ of 1:1 ammonia solution into a 50 cm³ standard flask* make up the solution to the mark with distilled water and mix well for uniform concentration.

Switch on the colorimeter and warm it for 5 minutes. Using the filter knob (B), select the wave length or λ_{max}=620 nm for copper sulphate solution. Take the blank solution in the cell tube and keep it in the colorimeter and adjust zero absorbance for 100% transmittance using the knob (A). Then measure the optical density or absorbance for each standard solution and test solution using a photo electric colorimeter. A straight line graph is obtained by plotting optical density or 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.

$$CuSO_4 + 4NH_3 \longrightarrow [Cu(NH_3)_4SO_4] \text{ (blue color)}$$
$$\text{(Cuprammonium sulphate)}$$
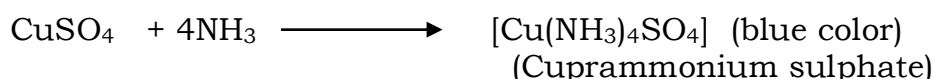
Preparation of blank solution: - Blank solution can be prepared by adding 5.0 cm^3 of 1:1 ammonia and distilled water in 50 cm^3 standard flask. Mix well for uniform concentration.

Results:

1. The volume of test solution given   = _____ cm^3

2. Absorbance of test solution   = _____

3. Concentration of copper in test solution   = _____mg/ cm^3

4. Concentration of copper sulphate solution = _____ g/cm^3

OBSERVATIONS AND CALCULATIONS

Estimation of acid mixture.

Vol. of NaOH in cm³	Conductance Ohm⁻¹ cm⁻¹	Vol. of NaOH in cm³	Conductance 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		10.5	

V_1=Volume of NaOH required to neutralize strong acid

V_2-V_1=Volume of NaOH required to neutralize weak acid

EXPERIMENT NO. 3   **Date:** ☐☐☐☐☐☐☐☐

CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE USING STANDARD SODIUM HYDROXIDE

THEORY:

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

Conductometric titration may be applied for the determination of acid & acids present in a mixture. In the titration of a mixture of acid with a strong base (sodium hydroxide), the conductance falls until the strong acid is neutralized completely, and the weak acid undergo dissociate and gets neutralized. This results in the increases in conductance of the solution as the weak acid is consumed and converted into salt. When the neutralization of second acid is complete, there is a steep increase in conductance due to ions provided by the strong base.

PROCEDURE:

Transfer the given acid mixture (25 cm^3) into a clean 100 cm^3 beaker. Fill standard NaOH (0.2N) solution into 10 cm^3 burette. Place the conductivity cell inside the beaker, so that the electrodes are immersed completely in the acid solution. Connect the cell to the conductometer and measure the conductance. Add 0.5 cm^3 of NaOH each time, till there is a rise in conductance. After each addition stir the solution and note down the corresponding decrease in the conductivity of the solution. As the titration proceeds the conductance is gradually a decreases and then increases slowly and finally increases sharply. Take 5-6 readings when conductance begins to increase sharply. Plot a graph of conductance against volume of NaOH added.

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Estimation of HCl

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

$$\text{Normality of HCl} = \frac{N_{NaOH} \times V_1}{25}$$

$$= \frac{0.2 \times \text{.....................}}{25} \quad = \text{................}N$$

Weight per liter of HCl = X = N$_{HCl}$ X Equivalent weight of HCl

$$= \text{--------------------} X\ 36.5$$

$$X = \text{--------------------}g.$$

Estimation of CH$_3$COOH

Volume of NaOH required to neutralize CH$_3$COOH = $(V_2 - V_1)$ =..............cm^3

$$\text{Normality of CH}_3\text{COOH} = \frac{N_{NaOH} \times V_{(V2-V1)}}{25}$$

$$= \frac{0.2 \times \text{.....................}}{25} \quad = \text{................}N$$

Weight per liter of CH$_3$COOH = X = N$_{CH3COOH}$ X Equivalent weight of CH$_3$COOH

$$= \text{--------------------} X\ 60.05$$

$$X = \text{--------------------}g.$$

From the graph determine the volume of NaOH required be V_1 cm^3 for strong acid. As the titration proceeds, the second neutralization point V_2 will be obtained. The Difference V_2-V_1 will give the amount of base neutralization for weak acid. Calculate the weight per liter of HCl and CH$_3$COOH.

HCl + NaOH $\longrightarrow$ NaCl + H$_2$O

CH$_3$COOH+NaOH $\longrightarrow$ CH$_3$COONa + H$_2$O
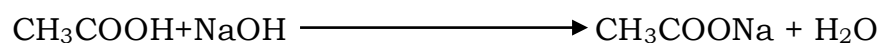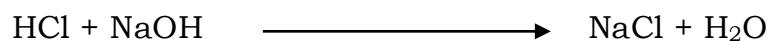
RESULT: a) The weight of HCl/dm^3 _____ g

b) The weight of CH$_3$ COOH/dm^3 _____ g

OBSERVATIONS AND CALCULATIONS

Volume of NaOH (cm³)	pH	ΔPH	ΔV	ΔPH / ΔV
0.0				
0.5				
1.0				
1.5				
2.0				
2.5				
3.0				
3.5				
4.0				
4.5				
5.0			0.5	
5.5				
6.0				
6.5				
7.0				
7.5				
8.0				
8.5				
9.0				
9.5				
10.0				

RESULTS:

1. Equivalence Point   =_____

2. Half E.P   =_____

3. pH at E.P   =_____

4. pH at H.E.P.   =_____

5. pKa of the given acid   =_____

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EXPERIMENT NO. 4   **Date:** ☐☐☐☐☐☐☐☐

DETERMINATION OF pKa VALUE OF A WEAK ACID USING pH METER

THEORY:

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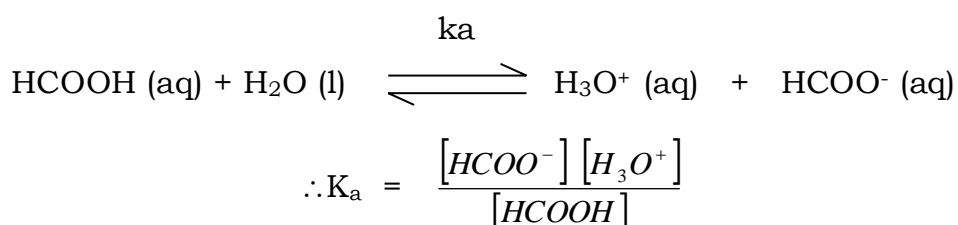are completely ionized in aqueous solution and hence it is impossible to determine the dissociation constant of strong electrolytes. But in case of weak electrolytes are partially ionized in aqueous solution and hence the equilibrium constant of weak electrolytes can be experimentally determined.

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

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

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

pK_a is a modern method of expressing the strength of weak acid ,
ie., $pKa = - \log_{10} Ka$

pK_a 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 changes in pH against different volume of the base added and from the graph, we obatin the equivalence point of an acid. The pKa value of an acid can be calculated using Henderson equation,

$$pH = pKa + \log_{10} \frac{\left[Salt\right]}{\left[Acid\right]}$$

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 = _____ cm^3

Y –axis, 1.0 cm = _____ pH

Graph No.1

HEP   **EP**

Volume of NaOH added in cm^3

Scale :

X –axis, 1.0 cm = _____ cm^3

Y –axis, 1.0 cm = _____ units

$\dfrac{\Delta pH}{\Delta V}$

Graph No.2

EP

Volume of NaOH added in cm^3

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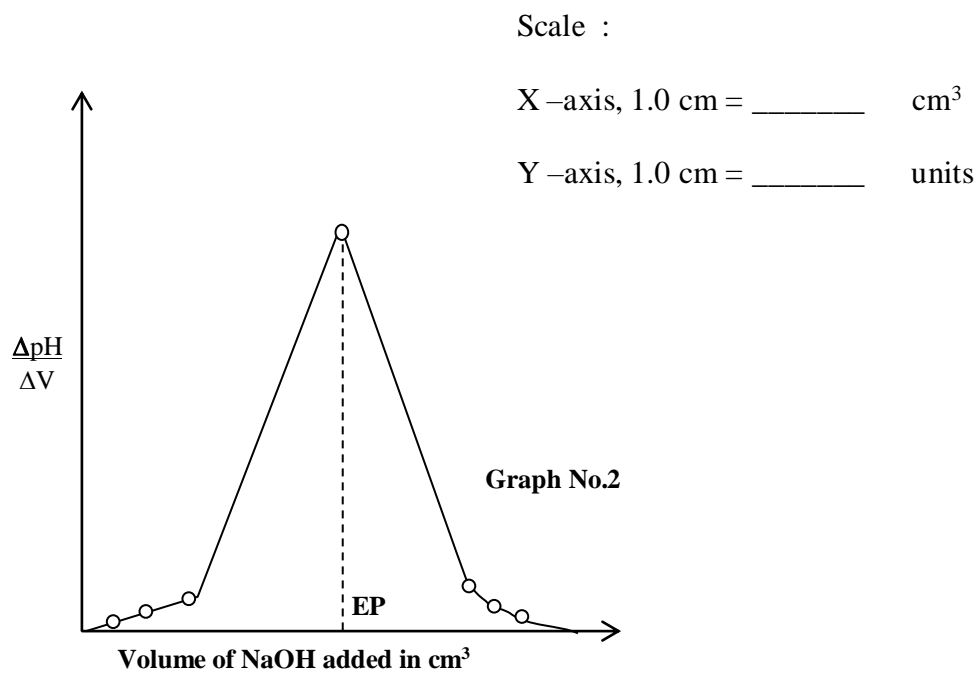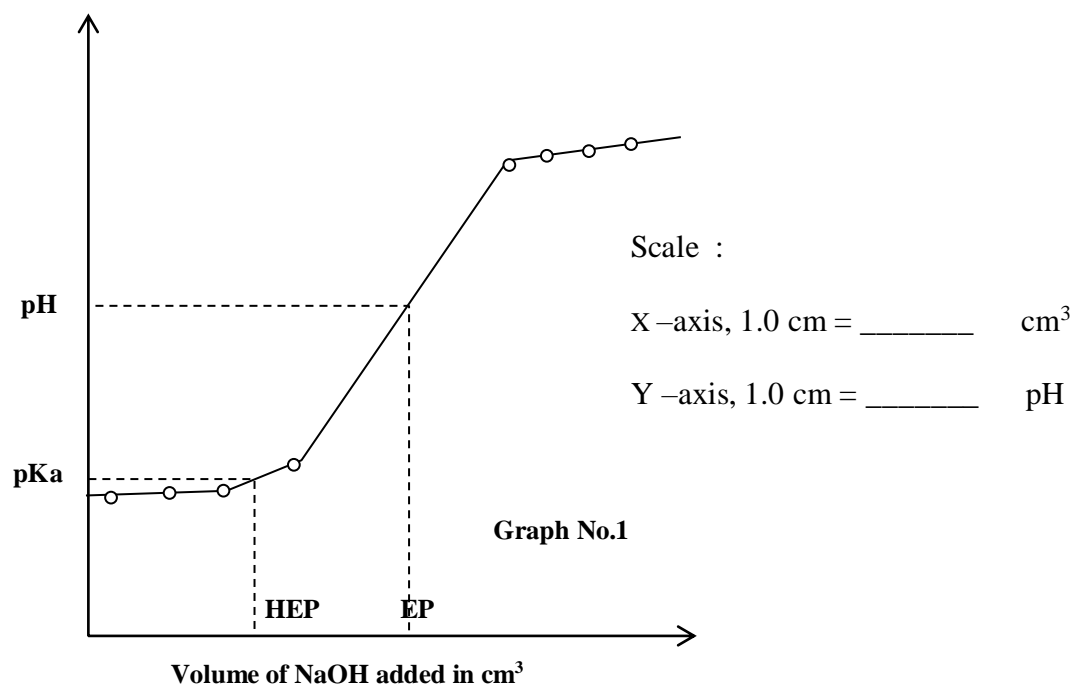
PROCEDURE:

Transfer given quantity of weak acid (25 cm^3) into a clean 100 cm^3 beaker followed by adding one t.t. of distilled water.

Switch on the pH meter and adjust the temperature knob to the lab temperature. Connect the combined electrode (i.e. glass and calomel electrode) to the pH meter. Wash the electrode with distilled water and immerse the electrode into the acid taken in a beaker. Note down the pH of the acid. Add 0.5 cm^3 of sodium hydroxide solution at a time taken in a micro burette in to the acid, stir the solution using magnetic stirrer and measure the pH of the acid and continue the addition of sodium hydroxide until the sudden raise in the pH. Then take few more readings and plot the graph, pH against the volume of sodium hydroxide added (graph No1). 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.2) and from the graph 2, determine the equivalence point of the acid. Determine the half equivalence point that gives the pKa value of weak acid by the graph 1.

Note: Switch off the instrument after experiment is completed.

RESULTS:

1. Equivalence Point   =_____

2. Half E.P   =_____

3. pH at E.P   =_____

4. pH at H.E.P.   =_____

5. pKa of the given acid   =_____

OBSERVATIONS AND CALCULATIONS

Trial No.	I	II	III	Average time in sec
Time taken by water in sec (t_W)				
Time taken by liquid in sec (t_L)				

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

Note: lab temperature=..........°C

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

$$\eta_L = \frac{\eta_w \, t_L d_L}{t_w d_w} = \text{_____ milli poise}$$

∴ η_L = co – efficient of viscosity of liquid = _____ milli poise

EXPERIMENT NO.5   **Date:**

DETERMINATION OF VISCOSITY COEFFICIENT OF 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 to maintain 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, $\eta = \dfrac{\pi P r 4 t}{8 V l}$, 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, $\dfrac{\eta_w}{\eta_L} = \dfrac{t_w d_w}{t_L d_L}$

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. 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.

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Upper mark (**A**)

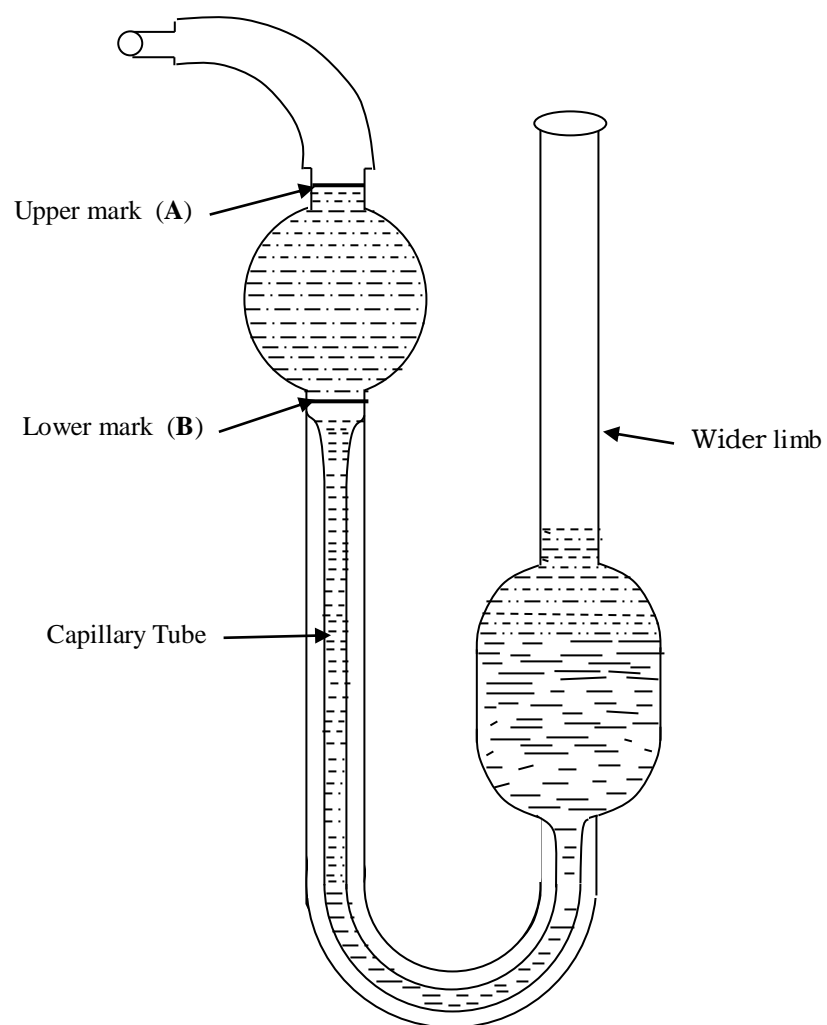Lower mark (**B**)

Capillary Tube

Wider limb

Fig. Viscometer

PROCEDURE:

1. Clean the given viscometer with distilled water and rinse with acetone. Dry it in an electric oven and then fix the viscometer vertically to a stand.

2. Transfer a known volume (say 15 cm³) of distilled water taken in a burette into the viscometer. Suck the distilled water above the upper mark 'A' in the viscometer and allow it to flow freely through a capillary. Start the stop clock when the water level just crosses the upper mark '**A**' and stop it when the water level just crosses the lower mark '**B**' in the viscometer. Note down the time of flow of water in seconds (t_W) and repeat the experiment to get agreeing values.

3. Pour out the distilled water and rinse the viscometer with acetone again. Dry it and fix the viscometer vertically to a stand.

4. Transfer the known volume (say 15 cm³) of the given organic liquid into a viscometer. Note down the time of flow of organic liquid (t_L) in seconds as similar to the water and repeat the experiment to get agreeing values.

5. 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.

RESULT: ∴ η_L = co – efficient of viscosity of liquid = _____ milli poise

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.

PART-B

VOLUMETRIC

ANALYSIS

OBSERVATIONS AND CALCULATIONS:

1. Preparation of standard solution of disodium salt of EDTA

Weight of weighing bottle + EDTA salt   = _____g.

Weight of empty weighing bottle   = _____g.

∴ Weight of EDTA salt taken   = _____g.

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

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

2. Determination of total hardness of water.

Burette   : Standard EDTA solution

Conical flask   : 25 cm^3 of hard water + 5cm^3 of NH_4OH – NH_4Cl buffer .

Indicator   :   Eriochrome black – T

End point   : Wine red to clear blue colour

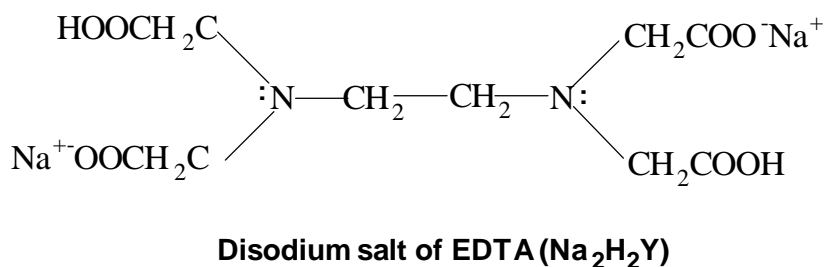| Burette readings(cm^3)	I	II	III
Final readings			
Initial readings			
Volume of EDTA run down			

∴ Volume of EDTA required V= _____cm^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.

$$HOOCH_2C \diagdown$$
$$\qquad :N—CH_2—CH_2—N: \qquad$$
$$HOOCH_2C \diagup \qquad\qquad\qquad \diagdown CH_2COOH$$
$$\qquad\qquad CH_2COOH$$

EDTA(H_4Y)

$$HOOCH_2C \diagdown \qquad\qquad\qquad CH_2COO^-Na^+$$
$$\qquad :N—CH_2—CH_2—N:$$
$$Na^+{}^-OOCH_2C \diagup \qquad\qquad \diagdown CH_2COOH$$

Disodium salt of EDTA (Na_2H_2Y)
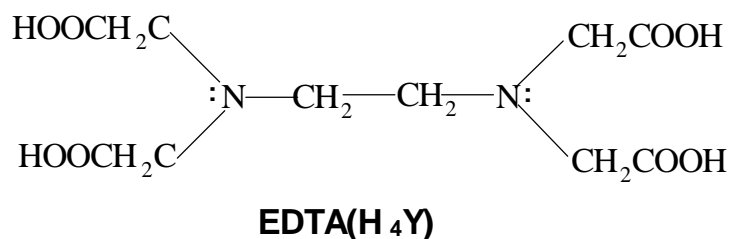
NOTE: Molecular weight of CaCO$_3$ = 100

1000 cm^3 of 1M of EDTA = one g molecular weight of CaCO$_3$ (100)

$\therefore$ V cm^3 of (a) M EDTA = X $= \dfrac{\text{(a) x V x 100}}{1000}$ =

$\qquad\qquad\qquad\qquad$ X   = _____g of CaCO$_3$

$\therefore$ Weight of CaCO$_3$ present in 25 cm^3 of hard water = X = _____g

$\therefore$ Weight of CaCO$_3$ present in 25 cm^3
of hard water in terms of ppm $\qquad$ = Y = $\dfrac{\text{X x } 10^6}{25}$

$\qquad\qquad$ Y= $\dfrac{\text{x } 10^6}{25}$ = _____ppm

Thus total hardness of water in terms of ppm   = Y =_____ppm

Note: Parts per million: ppm is the parts of CaCO$_3$ equivalent hardness present per million **or** 10^6 parts of water.

ie: 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 given disodium salt of EDTA accurately using an electronic balance. Transfer the crystals of EDTA into a clean 250 cm³ standard flask using a funnel. To this add **one** test tube of ammonium hydroxide and little distilled water and shake well to dissolve the crystals. 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 25cm³ of the given hard water sample into a clean conical flask, to this add 5 cm³ of ammonia-ammonium chloride buffer solution, and 2 drops of Eriochrome black–T indicator. Titrate this mixture against standard EDTA solution taken in a burette until the wine red color changes to clear 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.

REACTIONS:

$M^{2+}(Ca^{2+}$ or $Mg^{2+})$ + In $\longrightarrow$ M – In (wine red color)
(hard water)   Indicator   (metal ion – In complex)

M – In + Na_2H_2Y $\longrightarrow$ M – Y + 2 Na⁺ + 2H⁺ + In
(blue)

(Metal ion – EDTA complex)
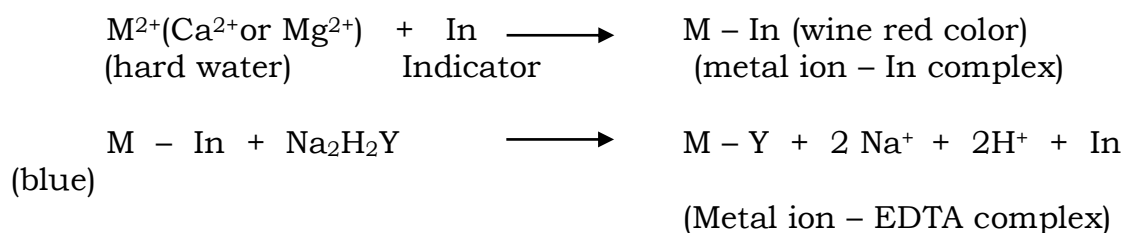
RESULT: The total hardness of the given water sample is..............ppm of CaCO₃.

OBSERVATIONS AND CALCULATIONS:

I . Preparation of standard solution of disodium salt of EDTA

Weight of weighing bottle + EDTA salt   = _____g.

Weight of empty weighing bottle   = _____g.

$\therefore$ Weight of EDTA salt taken   = _____g.

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

$$M_{EDTA} \;=\; \frac{x \; 4}{372.24} \;=\; \underline{\hspace{2cm}} \text{ (a) M}$$

II. Estimation of CaO in cement solution

Burette   : Standard EDTA solution

Conical flask   : 25 cm^3 of cement solution + 5cm^3 of 1:1 glycerol

                        + 5cm^3 of di-ethylamine + 10 cm^3 of 4N NaOH .

Indicator   : Pattern - Reeder's indicator

End point   : Wine red to purple blue color

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of EDTA run down in cm^3			

$\therefore$ Volume of EDTA required = V= _____ cm^3

EXPERIMENT NO. 2   **Date:**

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

THEORY:

Cement contains oxides of calcium, aluminum, 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 the given disodium salt of EDTA accurately using an electronic balance. Transfer the crystals of EDTA into a clean 250cm³ standard flask using a funnel. Dissolve the crystals by adding **one** 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.

NOTE: Molecular weight of CaO = 56.08

1000 cm^3 of 1M of EDTA = one gm molecular weight of CaO (56.08)

$\therefore$V cm^3 of (a) M EDTA = X = $\dfrac{(a) \times V \times 56.08}{1000}$ = $\dfrac{x \quad\quad\quad \times 56..08}{1000}$

X = _____ g of CaO

$\therefore$ Weight of CaO present in 25 cm^3 of cement solution   = X = _____g

$\therefore$ Weight of CaO present in 1000cm^3 of cement solution = Y = $\dfrac{X \times 1000}{25}$

Y = $\dfrac{\rule{3cm}{0.4pt}}{}$

Y = _____g

Result:
Amount of CaO in the 1000 cm^3 cement solution= _____g

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II. Estimation of calcium oxide:

Pipette out 25cm³ of the given cement solution into a clean conical flask. Add 5cm³ of 1:1 glycerol, 5cm³ of diethyl amine and 10cm³ of 4N sodium hydroxide. Add 3-4 drops of Pattern- Reeder's indicator and titrate this against EDTA solution taken in a burette until wine red color 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:

$$(CaO + SiO_2) + 2HCl \longrightarrow CaCl_2 + SiO_2 \downarrow + H_2O$$
Cement

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

$$Ca – In + EDTA \longrightarrow Ca – EDTA \ complex + In \ (blue \ color)$$
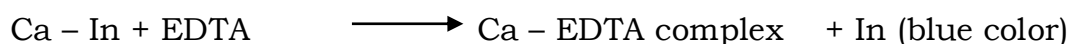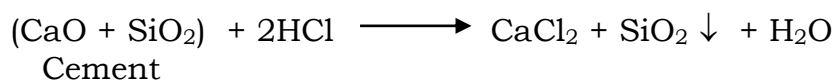
RESULT: The amount of CaO present in the given cement solution is

.... g

OBSERVATIONS AND CALCULATIONS:

I. Preparation of brass solution:

Weight of brass taken is =…………………………g (W)

II. Estimation of copper:

Burette   : Standard hypo(sodium thiosulphate) solution

Conical flask   : 25 cm^3 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 + one t.t. of distilled water.

Indicator   : 2 cm^3 of freshly prepared starch.

End point   : Disappearance of blue color

Burette readings	I	II	III
Final readings			
Initial readings			
Volume of hypo run down in cm^3.			

∴ Volume of hypo required = V= _____ cm^3

NOTE: **1. Normality of hypo = 0.05 N**

     2. One gram equivalent weight of copper = 63.54

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 and excess of nitric acid destroyed by boiling with urea. The solution is neutralized 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 liberated iodine 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 which is commonly known as hypo.

PROCEDURE:

I. Preparation of Brass solution:

Weigh the given brass pieces accurately weigh and transfer it into a clean conical flask. Add one t.t. of 1:1 nitric acid. Warm gently to dissolve the brass piece completely and add two t.t of distilled water. Add pinch 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 cm^3 standard flask. Make up the solution to the mark by adding distilled water and mix well for uniform concentration.

$\therefore$ 1000 cm³ of 1 N of hypo = one g equivalent weight of copper (63.54)

$\therefore$ V cm³ of 0.05 N hypo = X = $\dfrac{\text{V x 0.05 N x 63.54}}{1000}$ =---------------------

$$X = \underline{\hspace{2cm}} \text{ g of copper}$$

$\therefore$ Weight of copper present in 25 cm³ of brass solution = X = ---------------g

$\therefore$ Weight of copper present in 250 cm³ of brass solution = Y = $\dfrac{\text{X x 250}}{25}$

$$Y = \underline{\hspace{2cm}} \text{ g.}$$

$\therefore$ Percentage of copper in the

given brass sample = $\dfrac{\text{Y x 100}}{\text{Weight of brass taken (W)}}$
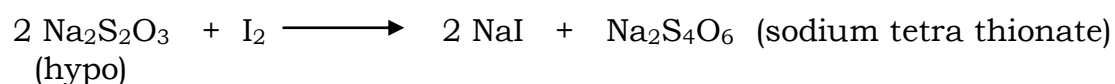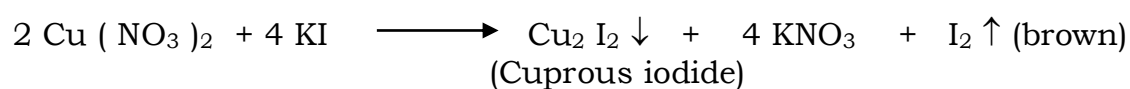
= --------------------------------------

$\therefore$ Percentage of copper in the given brass sample = -------------%

II Estimation of Copper:

Pipette out 25 cm³ 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 1/4th t.t. of acetic acid in excess. Add one t.t. of 10% potassium iodide solution and shake well. The liberated iodine titrated against standard sodium thiosulphate solution taken in a burette using starch as indicator towards the end point. (add starch when brown solution changes to light 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:

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

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

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

$$I_2 + starch \longrightarrow \text{Starch-iodide complex (deep blue)}$$
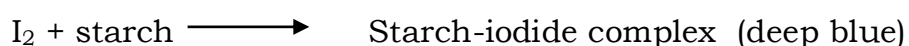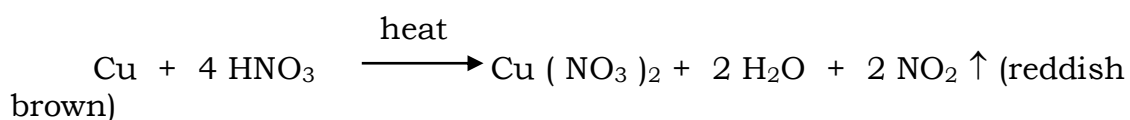
RESULT:
The percentage of Cu present in the given brass sample is...........%

OBSERVATIONS AND CALCULATIONS:

I. Preparation of standard potassium dichromate solution :

Weight of weighing bottle + $K_2Cr_2O_7$ crystals = _____g.

Weight of empty weighing bottle = _____g.

$\therefore$ Weight of $K_2Cr_2O_7$ crystals taken = _____g.

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

$$N_{K_2Cr_2O_7} \quad = \quad \frac{\text{x 4}}{49} = \ldots\ldots\ldots\ldots N(a)$$

II. Estimation of iron:

Burette : Standard $K_2Cr_2O_7$ solution

Conical flask : 25 cm^3 of haematite ore solution + ¼ t.t concentrated HCl + heat + SnCl$_2$ drop wise till yellow color changes to colorless + 5-6 drops of SnCl$_2$ in excess + cool the solution to room temperature + 10 cm^3 of saturated HgCl$_2$ + shake well + one t.t. of distilled H$_2$O

Indicator : potassium ferri cyanide as external indicator

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

Burette readings	pilot	I	II	III
Final readings				
Initial readings				
Volume of K$_2$Cr$_2$O$_7$ run down in cm^{3}s				

$\therefore$ Volume of $K_2Cr_2O_7$ required = V = _____ cm^3

EXPERIMENT NO. 4   **Date:** ☐☐☐☐☐☐☐☐

DETERMINATION OF IRON IN A GIVEN SAMPLE OF HAEMATITE ORE SOLUTION

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 cm^3 and is supplied for the estimation. Iron is present in the solution as Fe^{3+} (ferric)ions and 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 mercuric chloride solution. The ferrous ions in the resulting solution is titrated against standard potassium dichromate using potassium ferricyanide as external indicator. Potassium ferricyanide produces an intense deep blue color with ferrous ions due to the formation of ferriferrocyanide complex.

PROCEDURE:

a) Preparation of standard solution of potassium dichromate:

Weigh given potassium dichromate crystals accurately using an electronic balance. Transfer the crystals of potassium dichromate into a clean 250cm^3 standard flask using a 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.

NOTE: **Equivalent weight of iron** = **55.85 g**

1000 cm³ of 1 N of $K_2Cr_2O_7$ = one gm equivalent weight of iron (55.85g)

$$\therefore \text{V cm}^3 \text{ of (a) N } _{K_2Cr_2O_7} = X = \frac{(a) \times V \times 55.85}{1000}$$

$$X = \frac{}{1000} = \underline{}gm \text{ of iron}$$

$\therefore$ Weight of iron present in 25 cm³ of haematite solution = X = _____g.

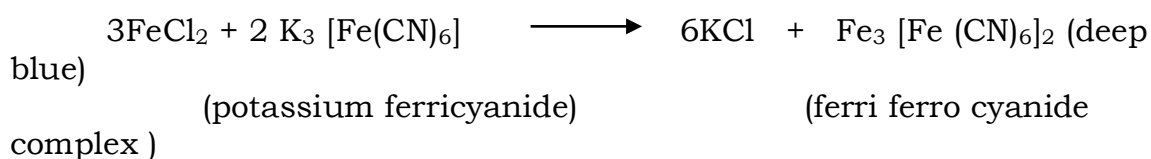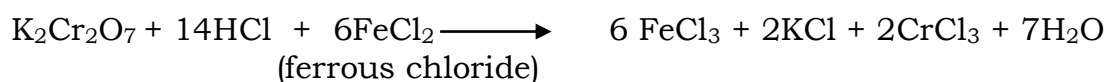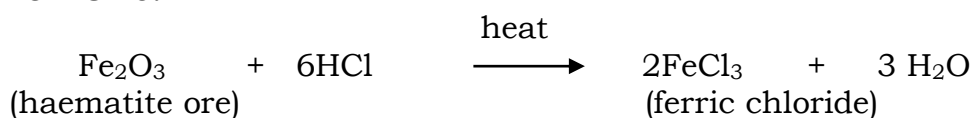$\therefore$ Weight of iron present in 1000 cm³ of

$$\text{Haematite ore solution} = Y = \frac{X \times 1000}{25} = $$

$$Y = \underline{}g.$$

b) Estimation of iron:

Pipette out 25cm^3 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 color solution becomes colorless and then add 6-8 drops of stannous chloride in excess. Cool the solution to lab temperature and add 10cm^3 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 amount of iron in the given sample of haematite ore solution.

REACTIONS:

$$Fe_2O_3 + 6HCl \xrightarrow{heat} 2FeCl_3 + 3H_2O$$
(haematite ore)   (ferric chloride)

$$K_2Cr_2O_7 + 14HCl + 6FeCl_2 \longrightarrow 6FeCl_3 + 2KCl + 2CrCl_3 + 7H_2O$$
(ferrous chloride)

$$3FeCl_2 + 2K_3[Fe(CN)_6] \longrightarrow 6KCl + Fe_3[Fe(CN)_6]_2 \text{ (deep blue)}$$
(potassium ferricyanide)   (ferri ferro cyanide complex)

RESULT:

The amount of iron in the given Haematite Ore solution is............ g

OBSERVATIONS AND CALCULATIONS:

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

Weight of weighing bottle + FAS crystals   =_____g.

Weight of empty weighing bottle   =_____g.

∴ Weight of FAS crystals taken   =_____g.

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

$$N_{FAS} = \frac{ \text{x 4}}{392} = \underline{} \text{(a) N}$$

II. Estimation of Chemical Oxygen Demand(back titration)

Burette   : Standard FAS solution

Conical flask   : 25 cm³ of waste water +25 cm³ of standard $K_2Cr_2O_7$ solution + one tt of 1:1 H_2SO_4 .

Indicator   : 4 – 6 drops of ferroin indicator

End point   : Blue green to reddish brown color

Burette readings	I	II	III	
Final readings				
Initial readings				
Volume of FAS run down in cm³				

∴ Volume of FAS solution required = V_1 = _____ cm³

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 oxidizing 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 oxdisable materials. They oxidize 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 oxidized partially by this procedure. This difficulty is overcome by adding mercuric sulphate to the sample.

PROCEDURE:

I. PREPARATION OF STANDARD MOHR'S SALT OR FERROUS AMMONIUM SULPHATE (FAS) SOLUTION:

Weigh given Mohr's salt crystals accurately using an electronic balance and transfer the crystals into a clean 250 cm³ standard flask. Dissolve the crystals by adding two t.t. of dilute sulphuric acid and little distilled water. and make it up 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 cm^3 of $K_2Cr_2O_7$ solution + 1tt of 1:1 H_2SO_4

   Indicator   :   4 – 6 drops of Ferrion indicator

   End Point   :   Blue green to reddish brown color

$\therefore$Volume of FAS solution required for blank titration

$\qquad\qquad\qquad = \mathbf{V_2}$ = Final B.R - Initial B.R=.....................

$\qquad\qquad\qquad V_2$=_____cm^3

$\therefore$Volume of $K_2Cr_2O_7$ required for the oxidation
  of organic compounds of waste water = $\mathbf{V_2 – V_1}$ = _____= _____ cm^3

1000 cm^3 of 1 N of FAS = 1000 cm^3 of 1N $K_2Cr_2O_7$ solution = 8 g of oxygen

$V_2 – V_1$ cm^3 of (a) N FAS = X = $\dfrac{8 \times (a) \times V_2 - V_1}{1000}$ =

$\qquad\qquad X = \dfrac{8 \times \rule{3cm}{0.4pt}}{1000}$

$\qquad\qquad X =$ _____g of oxygen

$\therefore$ Amount of O_2 required to oxidise organic
compounds present in 25 cm^3 of waste water   = X = _____g.

$\therefore$ Amount of O_2 required to oxidize organic
 compounds present in 1000cm^3 of waste water = Y = $\dfrac{X \times 1000}{25}$ = _____

$\qquad\qquad\qquad Y =$ _____g.

$\qquad \therefore$ COD of waste water   = Y x 1000 = _____ x 1000 mg
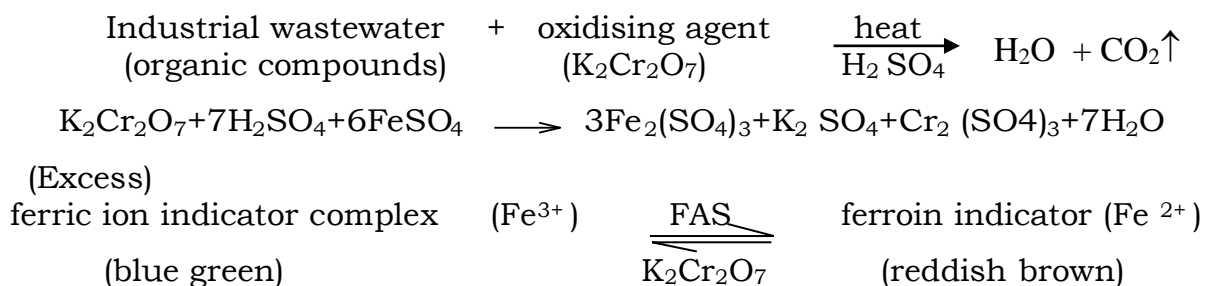$\therefore$ COD of waste water   = _____ mg of oxygen

II. Determination of COD of waste water:

Pipette out 25 cm^3 of the industrial waste water and add exactly 25cm^3 of standard potassium dichromate solution (0.1N) into a clean conical flask. Add one tt of 1:1 sulphuric acid and 2 to 4 drop of ferroin indicator and titrate against standard Mohr's salt solution taken in a burette until blue-green color 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 oxidize organic compounds and calculate the COD of the given industrial waste water.

III.   Blank titration

Add exactly 25cm^3 of standard potassium dichromate solution into a clean conical flask and one tt 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:

Industrial wastewater   +   oxidising agent $\xrightarrow[\text{H}_2\text{SO}_4]{\text{heat}}$ H_2O + $CO_2\uparrow$
(organic compounds)   ($K_2Cr_2O_7$)

$K_2Cr_2O_7 + 7H_2SO_4 + 6FeSO_4 \longrightarrow 3Fe_2(SO_4)_3 + K_2SO_4 + Cr_2(SO4)_3 + 7H_2O$
(Excess)

ferric ion indicator complex   (Fe^{3+}) $\underset{K_2Cr_2O_7}{\overset{FAS}{\rightleftharpoons}}$ ferroin indicator (Fe^{2+})
   (blue green)   (reddish brown)

RESULT:
The COD of the given waste water sample contains ....mg of oxygen

Determination of chloride content of water (Iodometric method)

a. *Principle:* Chlorine will liberate free iodine from potassium iodide (KI) solutions at pH 8 or less. The liberated iodine is titrated with a standard solution of sodium thiosulfate ($Na_2S_2O_3$) with starch as the indicator. Titrate at pH 3 to 4 because the reaction is not stoichiometric at neutral pH due to partial oxida- tion of thiosulfate to sulfate.

b. *Interference:* Oxidized forms of manganese and other oxidizing agents interfere. Reducing agents such as organic sulfides also interfere. Although the neutral titration mini- mizes the interfering effect of ferric and nitrite ions, the acid titration is preferred because some forms of combined chlo-rine do not react at pH 7. Use only acetic acid for the acid titration; sulfuric acid (H_2SO_4) will increase interferences; *never use hydrochloric acid (HCl)*. See Section A.3 for dis- cussion of other interferences.

2. Apparatus

For a description of the amperometric end-point detection apparatus and a discussion of its use, see D.2*a*.

3. Reagents

a. *Standard phenylarsine oxide solution,* 0.005 64*N*: Dissolve approximately 0.8 g phenylarsine oxide powder in 150 mL 0.3*N* NaOH solution. After settling, decant 110 mL into 800 mL distilled water and mix thoroughly. Bring to pH 6 to 7 with 6*N* HCl and dilute to 950 mL with distilled water. CAUTION: *Severe poison, cancer suspect agent.*

Standardization—Accurately measure 5 to 10 mL freshly stan- dardized 0.0282*N* iodine solution into a flask and add 1 mL KI solution. Titrate with phenylarsine oxide solution, using the amperometric end point (Method D) or starch solution (see B.2*e*) as an indicator. Adjust to 0.005 64*N* and recheck against the standard iodine solution; 1.00 mL = 200 µg available chlorine. (CAUTION: *Toxic—take care to avoid ingestion.*)

1

Standard sodium thiosulfate solution.

b. *Standard sodium thiosulfate solution,* 0.005 64N: Prepare by diluting 0.1N $Na_2S_2O_3$. For maximum stability of the dilute solution, prepare by diluting an aged 0.1N solution with freshly boiled distilled water (to minimize bacterial action) and add 4 g $Na_4B_4O_7$/L. To inhibit mold formation optionally add either 10 mg HgI_2 or 2 drops toluene per liter of solution. Standardize daily as directed in B.2c using 0.005 64N $K_2Cr_2O_7$ or iodate solution. Use sufficient volume of sample so that the final dilu- tion does not exceed 1 + 2. Use an automatic buret of a type in which rubber does not come in contact with the solution. 1.00 mL = 200 μg available chlorine.

c. *Potassium iodide,* KI, crystals.

d. *Acetate buffer solution,* pH 4.0: Dissolve 146 g anhydrous $NaC_2H_3O_2$, or 243 g $NaC_2H_3O_2 \cdot 3H_2O$, in 400 mL distilled water, add 480 g conc acetic acid, and dilute to 1 L with chlorine-demand-free water.

e. *Standard arsenite solution,* 0.1N: Accurately weigh a stop- pered weighing bottle containing approximately 4.95 g arsenic trioxide, As_2O_3. Transfer without loss to a 1-L volumetric flask and again weigh bottle. Do not attempt to brush out adhering oxide. Moisten As_2O_3 with water and add 15 g NaOH and 100 mL distilled water. Swirl flask contents gently to dissolve. Dilute to 250 mL with distilled water and saturate with CO_2, thus converting all NaOH to $NaHCO_3$. Dilute to mark, stopper, and mix thoroughly. This solution will preserve its titer almost in- definitely. (CAUTION: *Severe poison. Cancer suspect agent.*)

$$\text{Normality} = \frac{\text{g As2O3}}{49.455}$$

Standard iodine solution, 0.1N: Dissolve 40 g KI in 25 mL chlorine-demand-free water, add 13 g resublimed iodine, and stir until dissolved. Transfer to a 1-L volumetric flask and dilute to mark.
Standardization—Accurately measure 40 to 50 mL 0.1N ar- senite solution into a flask and titrate with 0.1N iodine solution, using starch solution as indicator. To obtain accurate results, insure that the solution is saturated with CO_2 at end of titration by passing current of CO_2 through solution for a few minutes just before end point is reached, or add a few drops of HCl to liberate sufficient CO_2 to saturate solution. Alternatively standardize against $Na_2S_2O_3$;

Optionally, prepare 0.1000N iodine solution directly as a standard solution by weighing 12.69 g primary standard resub- limed iodine. Because I_2 may be volatilized and lose from both solid and solution, transfer the solid immediately to KI as Specified above

Never let solution stand in open containers for extended periods.

Standard iodine titrant, 0.0282N: Dissolve 25 g KI in a little distilled water in a 1-L volumetric flask, add correct amount of 0.1N iodine solution exactly standardized to yield a 0.0282N solution, and dilute to 1 L with chlorine-demand-free water. For accurate work, standardize daily according to directions in ¶ 3g above, using 5 to 10 mL of arsenite or $Na_2S_2O_3$ solution. Store in amber bottles or in the dark; protect solution from direct sunlight at all times and keep from all contact with rubber.

      a. *Starch indicator:* See B.2e.
      b. *Standard iodate titrant,* 0.005 64N: Dissolve 201.2 mg primary standard grade KIO_3, dried for 1 h at 103°C, or 183.3 mg primary standard anhydrous potassium bi-iodate in distilled water and dilute to 1 L.
      c. *Phosphoric acid solution,* H_3PO_4, 1 + 9.
      d. *Phosphoric acid-sulfamic acid solution:* Dissolve 20 g NH_2SO_3H in 1 L 1 + 9 phosphoric acid.
      e. *Chlorine-demand-free water:* Prepare chlorine-demand-free water from good-quality distilled or deionized water by adding sufficient chlorine to give 5 mg/L free chlorine. After standing 2 d this solution should contain at least 2 mg/L free chlorine; if not, discard and obtain better-quality water. Remove remaining free chlorine by placing container in sunlight or irradiating with an ultraviolet lamp. After several hours take sample, add KI, and measure total chlorine with a colorimetric method using a nessler tube to increase sensitivity. Do not use before last trace of free and combined chlorine has been re- moved.

Distilled water commonly contains ammonia and also may contain reducing agents. Collect good-quality distilled or deion- ized water in a sealed container from which water can be drawn by gravity. To the air inlet of the container add an H_2SO_4 trap consisting of a large test tube half filled with 1 + 1 H_2SO_4 connected in series with a similar but empty test tube. Fit both test tubes with stoppers and inlet tubes terminating near the bottom of the tubes and outlet tubes terminating near the top of the tubes. Connect outlet tube of trap containing H_2SO_4 to the distilled water container, connect inlet tube to outlet of empty test tube. The empty test tube will prevent discharge to the atmosphere of

H_2SO_4 due to temperature-induced pressure changes. Stored in such a container, chlorine-demand-free water is stable for several weeks unless bacterial growth occurs.

. Preparation for titration:

1) Volume of sample—For chlorine concentration of 10 mg/L or less, titrate 200 mL. For greater chlorine concentrations, use proportionately less sample and dilute to 200 mL with chlorine- demand-free water. Use a sample of such size that not more than 10 mL phenylarsine oxide solution is required.

2) Preparation for titration—Measure 5 mL 0.005 64N phe- nylarsine oxide or thiosulfate for chlorine concentrations from 2 to 5 mg/L, and 10 mL for concentrations of 5 to 10 mg/L, into a flask or casserole for titration with standard iodine or iodate. Start stirring. For titration by amperometry or standard iodine,

also add excess KI (approximately 1 g) and 4 mL acetate buffer solution or enough to reduce the pH to between 3.5 and 4.2.

b. Titration: Use one of the following:

1) Amperometric titration—Add 0.0282N iodine titrant in small increments from a 1-mL buret or pipet. Observe meter needle response as iodine is added: the pointer remains practi- cally stationary until the end point is approached, whereupon each iodine increment causes a temporary deflection of the microammeter, with the pointer dropping back to its original position. Stop titration at end point when a small increment of iodine titrant gives a definite pointer deflection upscale and the pointer does not return promptly to its original position. Record volume of iodine titrant used to reach end point.

2) Colorimetric (iodine) titration—Add 1 mL starch solution and titrate with 0.0282N iodine to the first appearance of blue color that persists after complete mixing.

3) Colorimetric (iodate) titration—To suitable flask or casse- role add 200 mL chlorine-demand-free water and add, with agitation, the required volume of reductant, an excess of KI (approximately 0.5 g), 2 mL 10% H_3PO_4 solution, and 1 mL starch solution in the order given, and titrate immediately* with 0.005 64N iodate solution to the first appearance of a blue color that persists after complete mixing. Designate volume of iodate solution used as A. Repeat procedure, substituting 200 mL sam- ple for the 200 mL chlorine-demand-free water. If sample is colored or turbid, titrate to the first change in color, using for comparison another portion of sample with H_3PO_4 added. Des- ignate this volume of iodate solution as B.

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5. Calculation

a. Titration with standard iodine:

$$\text{mg Cl as Cl}_2/\text{L} = \frac{(A - 5B) \times 200}{C}$$

where:

A = mL 0.005 64N reductant,
B = mL 0.0282 N I$_2$, and
C = mL sample.

BGS INSTITUTE OF TECHNOLOGY
Department of Engineering Chemistry

LABORATORY RUBRICS

Programme	Course Code	Subject	Credits	L-T-P-TL	Assessment		Exam Duration
					SEE	CIA	
B.E	18CHEL16/26	Engineering Chemistry Lab	02	0-0-3-3	60	40	3Hrs

Maximum Marks: 40

Continuous Internal Evaluation	Excellent (80%-100%)	Good (80%-60%)	Average (40%-50%)
a. Observation write up and punctuality (05)	Students should write the experiments in the Observation book neatly and attend the labs regularly	Students should write the experiments in the Observation book and attend the labs.	Improper maintenance of observation books and being irregular to the labs.
b. Conduction of experiment and utput (10)	Students should conduct the experiments following the given procedure, plot the graph, perform calculation and show the accurate results with S.I unit.	Students should conduct the experiments following the given procedure, plot the graph and perform calculation with average results.	Improper conduction of experiments, graph plotting and results without S.I. unit.
c. Viva voce (05)	They should answer all the questions.	If they answer some of the questions.	If they doesn't answer the questions.
d. Record write up (10)	They should write records neatly, legibly and with suitable Tabular columns.	They should write records with suitable tabular columns	Improper/poor maintenance of record.
e. Internal Test (10)	Students should write the given experiments containing Formula, Tabular column, Nature of the graph, conduct the experiment and show the results with S.I. unit.	Students must write the given experiments, conduct the experiment and show the results.	If the student write the experiment but fails to conduct it.