

Experiment No. 1**Date:** ___ / ___ / _____**DETERMINATION OF p^{K_a} OF A WEAK ACID USING p^H METER****Aim: To determine p^{K_a} value of weak acid using digital p^H meter**

Principle: During the titration of an acid with a base, the p^H of the solution rises gradually at first, then more rapidly, until at the equivalence point, there is a very sharp increase in pH for every small quantity of base added, once pass the equivalence point, the p^H increases only slightly on addition of excess base. The titration curve obtained by plotting changes in p^H at different amounts of the base added is given in figure.

According to Henderson-Hasselbalch equation. $p^H = p^{K_a} + \log [\text{salt}] / [\text{acid}]$. Where K_a is dissociation constant of the acid and $p^{K_a} = -\log K_a$.

At half equivalence point, $[\text{salt}] = [\text{acid}]$. And hence $p^H = p^{K_a}$. Therefore p^H at half equivalence point gives the p^{K_a} of weak acid.

Procedure: Pipette out 25ml of the given weak acid into a beaker. Immerse the glass and calomel electrode assembly into it. Connect the electrodes to pH meter and measure the pH of acid. Add NaOH from the burette in increments of 0.5ml and measure the pH after each addition. Plot the graph of $\Delta p^H / \Delta V$ vs volume of NaOH and determine the equivalence point(X). Plot the another graph of pH vs volume of NaOH and determine the half equivalence point (X/2). Determine the p^{K_a} value at half equivalence point.

Result: The p^{K_a} value of a given weak acid is -----

Observation and Calculation:

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

Equivalence point, $X = \text{-----} \text{Cm}^3$

Half equivalence point, $X/2 = \text{-----} \text{Cm}^3$

p^H at half equivalence point = $p^{Ka} = \text{-----}$

p^{Ka} of given weak acid = -----

Experiment No. 2**Date:** ___ / ___ / _____**POTENTIOMETRIC TITRATION-ESTIMATION OF FAS USING STANDARD K₂Cr₂O₇ SOLUTION****Aim: Estimation of potentiometric titration-estimation of FAS using standard K₂Cr₂O₇ solution**

Principle: The procedure of using a measurement of emf to determine the concentration of ionic species in solution is called as potentiometry. The principle involved in potentiometric titration is the measurement of emf between two electrodes, an indicator electrode, (the potential of which is function of the concentration of the ion to be determined) and a reference electrode of constant potential. In this titration, the measurement of emf is made while the titration is in progress. The equivalence point of the reaction is revealed by a sudden change in potential in the plot of emf readings against the volume of titrant.

The determining factor in oxidation and reduction reactions is the ratio of the concentration of oxidized and reduced forms of certain species

$$E_{\text{cell}} = E^{\circ} + 0.0591/n \log [\text{Fe}^{3+}] / [\text{Fe}^{2+}]$$

Where E° = standard potential of the system.

The potential of the immersed electrode is controlled by the ratio of these concentrations. During the oxidation of a reducing agent or the reduction of an oxidizing agent, the ratio changes, hence the emf changes more rapidly in the vicinity of the end point.

Procedure:

Pipette out 25ml of the given FAS solution into a beaker. Add one test tube of dilute sulphuric acid. Immerse the platinum and calomel electrode assembly into it. Connect the electrodes to potentiometer and measure the potential. Add K₂Cr₂O₇ from the burette in increments of 0.5ml and measure the potential after each addition. Plot a graph of $\Delta E / \Delta V$ against volume of K₂Cr₂O₇ and determine the equivalence point. From the normality of K₂Cr₂O₇, calculate the normality and weight of FAS present in the given solution.

Result: Weight of FAS present in 25 cm³ of the given solution = ----- g

Observation and Calculation:

Volume of $K_2Cr_2O_7$ (V)	Emf (E) mv	ΔV	ΔE	$\Delta E / \Delta V$
0.0			-	-
0.5		0.5		
1.0		0.5		
1.5		0.5		
2.0		0.5		
2.5		0.5		
3.0		0.5		
3.5		0.5		
4.0		0.5		
4.5		0.5		
5.0		0.5		
5.5		0.5		
6.0		0.5		
6.5		0.5		
7.0		0.5		
7.5		0.5		
8.0		0.5		
8.5		0.5		
9.0		0.5		
9.5		0.5		
10.0		0.5		

$$N_{FAS} = \frac{N_{K_2Cr_2O_7} \times V_{K_2Cr_2O_7}}{\text{Volume of FAS}}$$

$$= \frac{0.5 \times}{25}$$

$$= \text{-----}$$

Weight of FAS present in a dm^3 of the given solution = $N_{FAS} \times \text{Eq. weight of FAS (392.14)}$

$$= \text{-----} \times 392.14 \text{ g}$$

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

Weight of FAS present in a 25 cm^3 of the given solution = $\frac{\text{-----}}{1000} \times 25$

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

Experiment No. 3

Date: ___ / ___ / _____

COLORIMETRIC ESTIMATION OF COPPER**Aim: Estimation of Copper colorimetrically****Principle:**

When a monochromatic light of intensity I_0 is incident on a transparent medium, a part I_a of it is absorbed, a part I_r is reflected & the remaining part I_t is transmitted.

$$I_0 = I_a + I_r + I_t$$

For a glass-air interface I_r is negligible.

$$\text{Therefore } I_0 = I_a + I_t$$

$I_t / I_0 = T$ called the transmittance $\log 1/T = \log I_0 / I_t$ is called the absorbance or optical density. The relation between absorbance A , concentration C & path length l is given by Beer-Lambert's law.

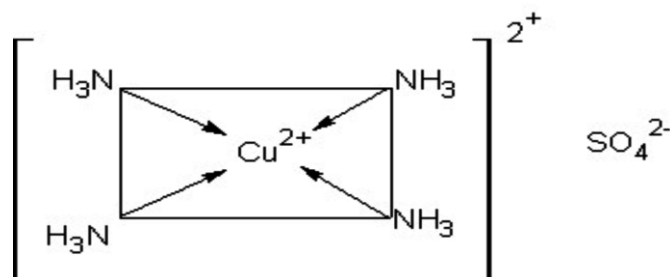
$$A = \log I_0 / I_t = \epsilon Ct$$

Where ' ϵ ' is the molar extinction coefficient, ' t ' is the path length. ' C ' is a constant for a given substance at a given wave length. If ' t ' is the path length is kept constant, then $A \propto C$. Hence a plot a graph of absorbance against concentration gives a straight line.

In a colorimeter, a narrow beam of light of proper wavelength passes through the test solution, towards a sensitive photocell (detector). Generally, a colorimeter is provided with arrangement of filter to select the light of required wavelength. The current generated in the photocell is proportional to the amount of light transmitted by the solution. This in turn depends upon the concentration of test solution. Thus, the current from the photocell will be more when the light transmitted by the solution is more. This generally happens when the test solution is more dilute.

A series of std. solutions of copper is treated with ammonia to get blue cuprammonium complex $[\text{Cu}(\text{NH}_3)_4]^{2+}$ and is diluted to a definite volume. The absorbance of each solution is measured at 620 nm, since the complex shows maximum absorbance at this wavelength. The absorbance values are plotted against volume of copper sulphate to get a calibration curve.

A known volume of the test solution is treated with ammonia & diluted up to the mark. The absorbance of this solution at 620 nm is measured & its concentration is determined from the calibration curve.



Procedure:

Transfer the given copper sulphate solution (stock solution) to a burette and draw out 2,4,6,8 & 10 ml of given copper sulphate solution into 5 separate 50 ml volumetric flasks. Now **collect the test solution from the teacher/examiner**, add **5ml of ammonia** solution to each of them and shake well and then dilute up to the mark with distilled water and mix well. Prepare a blank solution by diluting only 5 ml of ammonia solution in a 50 ml measuring flask up to the mark **with distilled water and mix well**. After 10min, **measure the absorbance of the solution against blank adjusting to 0.00 absorbance at 620nm** using a photoelectric colorimeter. Tabulate the readings as shown. Draw a calibration curve by plotting absorbance against volume of copper sulphate solution. Using the calibration curve, find out the volume of test solution and calculate the amount of copper in the given solution. (**Note: Use distilled water only to prepare the solutions**)

Result: The given test solution contains ----- mg of Cu

Observation and calculation :

Volume of copper Sulphate (ml)	Concentration of copper = 2.037mg x volume of copper sulphate solution	Absorbance (Optical density)
Blank Solution	-----	0.00
2.0	2.037mg x 2 =	
4.0	2.037mg x 4 =	
6.0	2.037mg x 6 =	
8.0	2.037mg x 8 =	
10.0	2.037mg x 10 =	
Test Solution	2.037mg x a =	

From graph, volume of the given test solution, a = _____ ml

Copper present in the given test solution = 2.037mg x volume of test solution ('a' ml)

$$= 2.037 \text{ mg x } \underline{\hspace{2cm}}$$

$$= \text{-----mg}$$

Experiment No. 4

Date: ___ / ___ / ____

**CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE USING
STANDARD NaOH SOLUTION.**

Aim: Estimation of acid mixture conductometrically using std. NaOH solution

Principle: Measurement of conductance can be employed to determine the end point in acid –base titrations. In conductometric titrations there is a sudden increase in conductance of the solution at the equivalence point. The principle underlying conductometric titrations is the substitution of ions of one mobility by ions of other mobility. Therefore, the conductance of a solution depends on the number & mobility of ions. The equivalence point is determined graphically by plotting conductance against titre values.

Conductometric titration may be applied for the determination of acid mixture. In this titration, on adding a strong base to a mixture of a strong acid and a weak acid, the conductance falls till a strong acid is neutralized completely due to removal of H^+ ions. The weak acid remains undissociated in the presence of strong acid. Once the strong acid is completely neutralized, the weak acid begins to dissociate and gets neutralized. This results in increase of conductance of solution. When the neutralization of second acid is complete, there will be a steep increase in conductance due to the ions furnished by strong base.

Procedure: Pipette out 25ml of acid mixture in a beaker. Immerse the conductivity cell in the given solution and connect it to conductivity meter. Stir the solution and measure the initial conductance of the solution. Add 1N NaOH from the burette in the increments of 0.5 ml after each addition stir the solution and note down the conductance. Continue the titration till decreasing trend changes to increasing trend.

Plot the graph of conductance on Y-axis and volume of NaOH on X-axis to get two straight lines. The point of intersection of two straight lines gives the equivalence point. The equivalence point corresponds to volume of NaOH required to neutralise HCl. By knowing the normality & volume of NaOH calculate the normality & amount of HCl & CH_3COOH .

Report:: The amount of HCl present in the given solution = _____ g

The amount of CH_3COOH present in the given solution = _____ g

Calculation:

Normality of NaOH = 1 N

Volume of NaOH required to neutralize HCl, $V_1 = \dots\dots\dots$ cm³

$$\text{Normality of HCl} = \frac{\text{Normality (NaOH)} \times \text{Volume of NaOH}}{\text{Volume of HCl}}$$

$$= \frac{1 \times \dots\dots\dots}{50}$$

$$= \dots\dots\dots \text{ N}$$

The weight of HCl /dm³ = Normality x Eq. Wt. of HCl

$$= \dots\dots\dots \times 36.5$$

$$= \dots\dots\dots \text{ g}$$

Volume of NaOH required to neutralize CH₃COOH, $V_2 - V_1 = \dots\dots\dots$ cm³

$$\text{Normality of CH}_3\text{COOH} = \frac{\text{Normality} \times \text{Volume of NaOH}}{\text{Volume of CH}_3\text{COOH}}$$

$$= \frac{1 \times \dots\dots\dots}{50}$$

$$= \dots\dots\dots \text{ N}$$

The weight of CH₃COOH /dm³ = Normality x Eq. Wt. of CH₃COOH

$$= \dots\dots\dots \times 60$$

$$= \dots\dots\dots \text{ g}$$

Volume of NaOH (ml)	Conductance (S/m)
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	
10.5	
11.0	
11.5	
12.0	
12.5	
13.0	
13.5	
14.0	
14.5	
15.0	
15.5	
16.0	
16.5	
17.0	
17.5	
18.0	
18.5	
19.0	
20.0	

Experiment No. 5**Date:** ___ / ___ / _____

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

Aim: To determine the viscosity coefficient of a given liquid using Ostwald's viscometer

Principle:

Viscosity arises due to internal friction between moving layer of molecules. A liquid flowing through cylindrical tube of uniform diameter is expected to move in the form of molecular layers. A layer close to surface is almost stationary, while that at the axis of the 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 between 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.

$$V = \frac{\Pi r^4 t}{8\eta l} \quad \text{Where } \rho = hgd$$

V = volume of the liquid, r = radius of the tube, l = length of the tube, ρ = pressure difference between two ends of the tube, η = the coefficient of viscosity of the liquid. If equal volumes of two different liquids are allowed to flow through the same tube under identical conditions then,

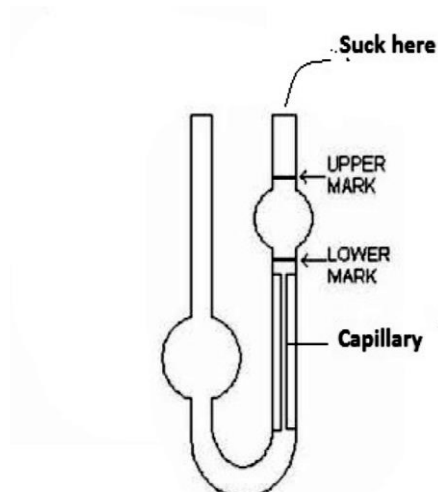
$$\eta_L = \frac{\eta_w \times t_l \times d_l}{t_w \times d_w}$$

The time t_L taken by the given liquid to travel through a certain distance in the tube is determined. The time t_w taken by water to travel through the same distance is measured. Knowing the densities d_L and d_w of the test liquid and water and also the coefficient of viscosity of water (η_w), coefficient of viscosity of the test liquid (η_L) can be calculated.

Procedure:

Pipette out 20 ml of given liquid into a wide limb of viscometer (fixed vertically to the stand) and suck the liquid through the other limb. Determine the time flow between 2 fixed points. One above & one below the bulb in the narrow limb of viscometer. Repeat and calculate the average time flow. Pour out the liquid, wash the viscometer with acetone and dry it. Now pipette out 20 ml of water into limb and determine the average time flow for water as before. Determine the coefficient of viscosity of the given liquid using the formula.

Result: The coefficient of viscosity of the given liquid = millipoise.

Observation and calculation :

	Trail-1 (sec)	Trail-2 (sec)	Trail-3 (sec)	Average (sec)
Liquid (t_L)				
Water (t_w)				

Laboratory Temperature =

η_w = Viscosity coefficient of water = millipoise

η_L = Viscosity coefficient of liquid = ?

d_w = density of water = g/cc

d_L = density of liquid = g/cc

$$\eta_L = \frac{\eta_w \times t_l \times d_l}{t_w \times d_w}$$

$$= \frac{\quad \times \quad}{\quad \times \quad}$$

$$= \frac{\quad}{\quad}$$

=-----millipoise.

The coefficient of viscosity of the given liquid = millipoise.

Experiment No. 6

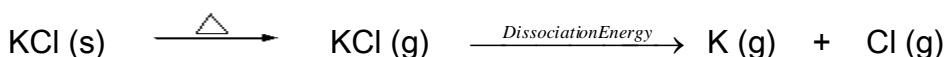
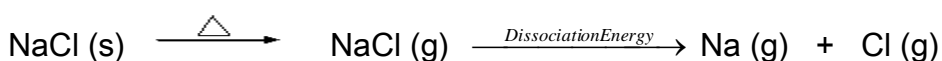
Date: ___ / ___ / _____

FLAME PHOTOMETRIC ESTIMATION OF SODIUM AND POTASSIUM.

Aim: To estimate the amount of sodium and potassium in the given sample of water using flame Photometer

Principle:

Flame photometry is an atomic emission technique used for detection of metals. If a solution containing metallic salts is aspirated into a flame, a vapor, which contains metallic atoms, will be formed. The electrons from the metallic atoms are then excited from ground state (E_1) to higher energy state (E_n) where $n = 2, 3, 4, \dots, 7$, by making use of thermal energy of flame. From higher energy states these electrons will return to the ground state by emitting radiations ($E_n - E_1 = h \nu$, where $n = 2, 3, 4, \dots, 7$), which are the characteristic of each element.



Flame photometer correlates the emitted radiations with the concentration of these elements. It is simple and rapid method for the elements that can be easily excited (Sodium, Potassium and other alkali metals)

A flame photometer is composed of the pressure regulator and flow meter for fuel gases an atomizer, burner, optical system, photosensitive detector and out put recorder. A filter of the element whose concentration is to be determined is inserted between the flame and the detector. Propane gas is used as fuel and air or oxygen is used as oxidant. Combination of these two will give a temperature of 1900°C The whole analysis depends on the flow rate of the fuel, oxidant, the rate of introduction of the sample and droplet size.

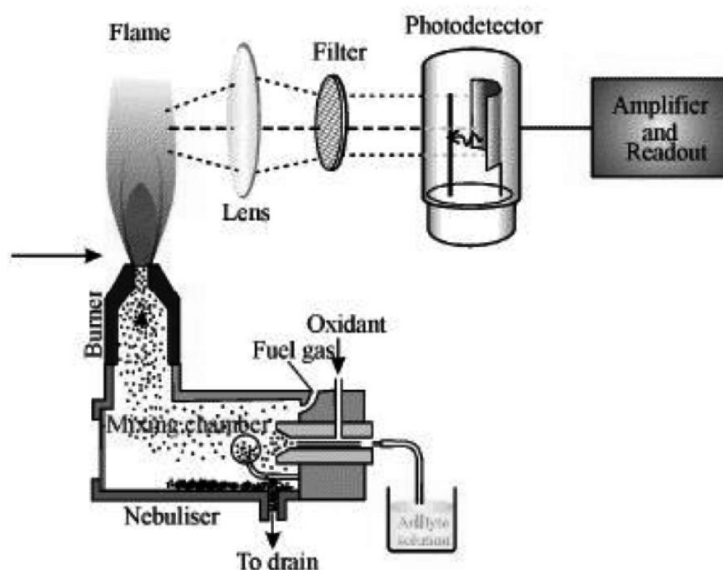


Fig. : Schematic diagram showing the layout of various components of a flame photometer

The sample containing the analyte is aspirated into the flame through atomizer. Radiation from resulting flame is collected by the lens and allowed to pass through an optical filter, which permits only the radiation characteristic of the element under investigation into the photocell. The output from the photocell represents the concentration and nature of the element.

Procedure:

Prepare the standard solution of sodium and potassium of concentration 2,4, 6, 8 & 10 ppm by transferring 2, 4, 6, 8, 10 ml of stock solution (50 ppm) into different 50ml volumetric flask using a burette. Dilute all the solutions up to the mark using distilled water, dilute the test solution of unknown concentration, and shake well for uniform concentration. Place the distilled water in the suction capillary of the instrument and set the instrument to read zero. Place each of the standard solutions in the suction capillary and set the instrument to read the flame emission intensity 2, 4, 6, 8 and 10 respectively using sodium filter (598 nm) (Rinse with distilled water after each reading) Dilute the given test solution up to the mark and mix well. Place the test solution in the suction capillary and record the reading. Draw a calibration curve by plotting the emission intensity on y-axis and volume of NaCl solution on X-axis. From the calibration curve, find out the volume of the given test solution and calculate the amount of sodium in the water sample.

Result: The given test solution contains ----- ppm of Na

Observation and calculation :

Volume of NaCl (ml)	Concentration of NaCl in ppm	Emission Intensity (ppm))
0.0	-----	
2.0	0.02	
4.0	0.04	
6.0	0.06	
8.0	0.08	
10.0	1.0	
Test solution		

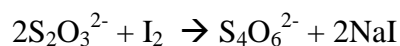
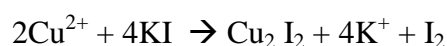
58.5g of NaCl contains 23.0g of Na

$$X \text{ ppm of NaCl contains (1000 ml)} = \frac{X \times 23}{58.5} = \underline{\hspace{2cm}} = \underline{\hspace{2cm}} \text{ ppm}$$

Experiment No. 7**Date:** ___ / ___ / _____**DETERMINATION OF PERCENTAGE OF COPPER IN BRASS USING STANDARD SODIUM THIOSULPHATE SOLUTION.****Aim: To determine the percentage of Copper in brass using Std. sodium thiosulphate solution*****Principle:***

The chief constituents of brass alloy are copper and zinc. It also contains small quantities of tin, lead & iron. The percentage composition of a typical brass alloy is Cu: 50 – 90 %, Zn: 20 -40 %, Sn : 0 – 6 % , Pb – 2 % , Fe : 0 – 1 %.

A solution of brass is made by dissolving the brass sample in minimum amount of nitric acid, excess of nitric acid is destroyed by boiling with urea. The solution is neutralized & made faintly acidic. Potassium iodide solution is added, the iodine liberated (cupric ions oxidizes iodide to iodine), is titrated against sodium thiosulphate using starch as indicator.

***Procedure:*****PART A: Preparation of brass solution**

Weigh out the given brass sample into a clean beaker. Add ¼ test tube con. HNO₃ till brass dissolves completely. Add 1 test tube of distilled water and 1 gm of urea, boil till all brown fumes are expelled. Cool to room temperature. Transfer the solution into a 250 ml volumetric flask and dilute up to the mark with distilled water and mix well.

PART B: Estimation of copper in brass solution

Pipette out 25 ml of brass solution into a clean conical flask; add ammonium hydroxide drop by drop until a pale blue precipitate is obtained. Dissolve the precipitate by adding 1/4th test tube of dilute acetic acid. Add 1 test tube of 10% KI solution. Titrate the liberated I₂ against standard Na₂S₂O₃ solution until the solution becomes pale yellow. Add 2 Cm³ of freshly prepared starch indicator, continue the titration till the blue color disappears leaving behind white precipitate. Repeat the titration till the concordant values are obtained.

RESULT: Percentage of Copper in the given brass sample = -----

Observation:**PART A: Preparation of brass solution**

Weigh out the given brass sample into a clean beaker. Add ¼ test tube con. HNO₃ till brass dissolves completely. Add 1 test tube of distilled water and 1 gm of urea, boil till all brown fumes are expelled. Cool to room temperature. Transfer the solution into a 250 ml volumetric flask and dilute up to the mark with distilled water and mix well.

PART B: Estimation of copper in brass solution

Burette reading	I	II	III
Final burette reading			
Initial burette reading			
Volume of Na ₂ S ₂ O ₃ run down (ml)			

Calculation:

Normality of Sodium thiosulphate $N_1 = 0.05 \text{ N}$

Weight of brass taken (w) = ----- g

Volume of Sodium thiosulphate consumed $V_1 = \text{----- } \text{Cm}^3$

1000 Cm^3 of 1 N Sodium thiosulphate = 63.54 g of Cu

Normality of Cu^{2+} solution, $N_2 = \frac{V_1 \times N_1}{25} =$

Weight of copper in a liter of the solution, $W_3 = \text{Normality of } \text{Cu}^{2+} \times \text{Eq. wt of Cu}$

$$= N_2 \times 63.54 =$$

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

Weight of copper in 250 ml of the solution, $W_4 = \frac{W_3}{4} =$

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

Percentage of copper in brass

$$= \frac{W_4 \times 100}{\text{Wt .of brass (w)}} =$$

$$= \text{.....}\%$$

Experiment No. 8

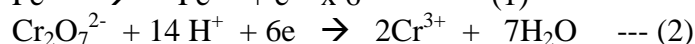
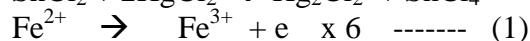
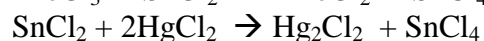
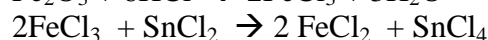
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**ESTIMATION OF IRON IN HAEMATITE ORE SOLUTION USING STANDARD
K₂Cr₂O₇ SOLUTION BY EXTERNAL INDICATOR METHOD.**

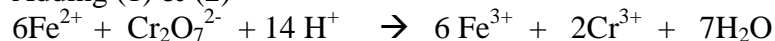
Aim: To determine the percentage of iron in the given sample of haematite ore solution by external indicator method

Principle:

Haematite is an important ore of iron containing mainly Fe₂O₃ and small amount of silica (SiO₂). Iron is present in the solution as Fe³⁺ ions. It is reduced to Fe²⁺ ions using stannous chloride in hot condition in presence of concentrated HCl. The excess of stannous chloride added is oxidized by treating the solution with mercuric chloride. The ferrous ions in the resulting solution are titrated against standard potassium dichromate using potassium ferricyanide as external indicator. Potassium ferricyanide produces an intense blue colour with ferrous ions due to the formation of ferriferrocyanide complex.



Adding (1) & (2)

**Procedure:****Part A: Preparation of standard K₂Cr₂O₇ solution**

Weigh out 0.5g of K₂Cr₂O₇ crystals accurately and transfer into a 250 ml volumetric flask. Dissolve in distilled water and dilute up to the mark, mix well.

Part B: Estimation of Iron

Pipette out 25 cm³ of the haematite ore solution into a clean conical flask. Add a quarter test tube of concentrated HCl and heat the solution to boiling. Add stannous chloride to the hot solution drop wise till the yellow solution turns colorless. Add 2 more drops to ensure complete reduction. Cool and add a quarter test tube of mercuric chloride (silky white ppt is formed). Add one test tube of water. Place number of drops of freshly prepared potassium ferricyanide indicator on the wax paper. Add a small quantity of K₂Cr₂O₇ from burette to the conical flask containing haematite ore solution and mix well. Take a drop of the solution from the conical flask and bring it in contact with a drop of the indicator on wax paper. The color of the indicator turns blue. Continue the titration by adding increment of 1cm³ of K₂Cr₂O₇ at a time and test till a drop of reaction mixture fails to produce any color with the indicator (Note: Clean the glass rod after every test). Repeat the titration by taking another 25 cm³ of the haematite ore solution. This time add most of the K₂Cr₂O₇ solution required at a stretch and then drop wise. Mix the contents of the flask after every addition and test a drop of the titrated mixture with a drop of the indicator as described above till the color of the indicator drop does not change. Repeat for concordant values.

RESULT: Percentage of iron in the given haematite ore solution = -----

Observation:**Part A: Preparation of standard $K_2Cr_2O_7$ solution**Weight of weighing bottle + $K_2Cr_2O_7$ crystals (W_1) = ----- gWeight of empty weighing bottle (W_2) = ----- gWeight of $K_2Cr_2O_7$ crystals transferred ($W_1 - W_2$) = ----- g

$$\text{Normality of } K_2Cr_2O_7 = \frac{(W_1 - W_2) \times 4}{\text{Equivalent weight of } K_2Cr_2O_7 (49)} = \text{-----}$$

$$= \text{-----}$$

Part B: Estimation of IronBurette : Std. $K_2Cr_2O_7$ SolutionConical Flask : 25 ml Haematite ore solution + $1/4^{\text{th}}$ test tube of con. HCl, boil +
SnCl₂ drop wise till colorless. Cool & add $1/4^{\text{th}}$ test tube of HgCl₂

Indicator : Potassium ferricyanide as external indicator.

Color Change: No change in the color of indicator

Burette reading	I	II	III
Final burette reading			
Initial burette reading			
Volume of $K_2Cr_2O_7$ run down in cm ³			

Calculation:Volume of $K_2Cr_2O_7$ consumed = -----Weight of Hematite ore in 25 cm³ = 0.1g1000 cm³ of 1N $K_2Cr_2O_7$ \equiv 55.85 g of Fe (1 equivalent of Fe)

$$\text{-----cm}^3 \text{ -----N } K_2Cr_2O_7 \equiv \frac{55.85 \times \quad \times}{1000 \times 1} =$$

 \equiv -----g of Fe / 25 Cm³ of haematite solution

$$\text{Percentage of iron in the given haematite ore solution} = \frac{\quad \text{g} \times 100}{\text{Weight of Ore (0.1)}} = \text{-----}$$

Experiment No. 9

Date: __ / __ / ____

ESTIMATION OF TOTAL HARDNESS OF WATER BY EDTA COMPLEXOMETRIC METHOD

Aim: To determine the total hardness of sample of water using Std. EDTA solution

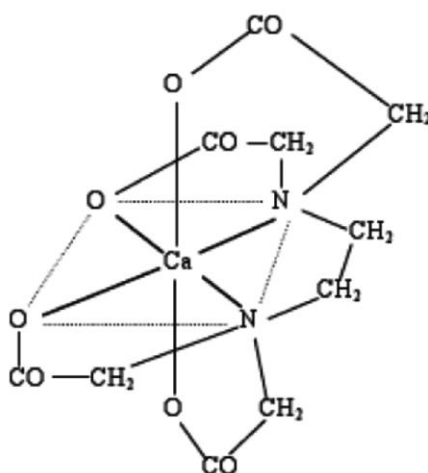
Principle:

Water with a high concentration of Mg^{2+} and Ca^{2+} is referred to as "hard" water, and the degree of hardness is linked to the concentrations of these two cations [public drinking water: 60-150 mg of $CaCO_3/L$]. Water with a low concentration of these ions is referred to as being "soft". Domestic water is "softened" by exchanging sodium ions for the calcium and magnesium ions.

The hardness of a water sample can be determined by titration of the alkaline earth cations with ethylene diamine tetra acetic acid (EDTA), a complexing agent. The acid itself is practically insoluble in water, so it is typical to use the disodium salt of EDTA (" Na_2H_2Y ").

As a complexing agent EDTA has the potential to bind at 6 sites (we say it is *hexadentate*). It quantitatively forms stable water soluble 1:1, complexes with metal ions (Mg^{2+} and Ca^{2+} : EBT-Mg complex is more stable than that of EBT-Ca complex). Thus EDTA can be used as a titrant to determine the hardness of water. EBT is used as the indicator, the end point being indicated by change in the color from the wine red to blue. Accurate determination of total hardness using EDTA/EBT depends on sharpening the transition from the wine red color of the EBT-complex to the blue color of the uncomplexed EBT.

When added to the solution containing metal ions buffered to a pH of about 10, EBT forms a wine red colored complex with the metal ions (see the below structure).



When the solution is titrated with EDTA, EDTA combines with the free metal ions (calcium ions in preference to magnesium ions) present in the solution. At the end point, when all the free metal ions are exhausted, added EDTA dissociates the wine red colored metal-EBT complex, consumes the metal ion and releases the blue colored free indicator. Hence the color changes from red wine to blue.

Procedure:**Part A: Preparation of standard solution of Disodium salt of EDTA**

Weigh out the given EDTA crystals accurately into a 250 ml volumetric flask. Add quarter test tube of ammonia. Dissolve in distilled water and dilute up to the mark, mix well.

Part B: Determination of total hardness of water sample

Pipette out 25 cm³ of the given water sample into a clean conical flask. Add 3 ml of Ammonia – ammonium chloride buffer and 1-2 drops of EBT indicator. Titrate against standard EDTA solution till the colour change from wine red to clear blue. Perform the titration slowly near end point. Repeat the titration to get concordant values

RESULT: The total hardness of given water sample = ----- ppm of CaCO₃

Observation:**Part A: Preparation of standard solution of Disodium salt of EDTA**

Weight of weighing bottle + EDTA salt (W₁) = ----- g

Weight of empty weighing bottle (W₂) = ----- g

Weight of EDTA transferred (W₁ – W₂) = ----- g

$$\text{Molarity of EDTA} = \frac{\text{Weight of EDTA taken (W}_1 - \text{W}_2) \times 4}{\text{Molecular weight of EDTA (372)}} = \text{-----}$$

$$= \text{----- M}$$

Part B: Determination of Total hardness of water sample

Burette reading	I	II	III
Final burette reading			
Initial burette reading			
Volume of EDTA run down (ml)			

Calculation:

Volume of EDTA consumed = -----

1000 cm³ of 1 M EDTA = 100 g of CaCO₃ (Molecular mass of CaCO₃ = 100)

-----ml of -----M EDTA = $\frac{X \times X \times 100}{1000 \times 1}$ g of CaCO₃ = -----

25 cm³ of the hard water sample contains ----- g of CaCO₃

10⁶ cm³ of the hard water sample contains = $\frac{X \times 10^6}{25}$ ppm of CaCO₃ = -----
 = ----- ppm of CaCO₃

Thus total hardness of given water sample = ----- ppm of CaCO₃

Experiment No. 10**Date:** ___ / ___ / _____**ESTIMATION OF CaO IN CEMENT SOLUTION BY RAPID EDTA METHOD.**

Aim: To determine the percentage of calcium oxide in the given sample of cement solution using Std. EDTA solution

Principle:

Cement contains compounds of calcium, aluminium, magnesium, iron and insoluble silica. When dissolved in acid, silica remains undissolved. On treating with ammonia, aluminium and iron can be precipitated as their hydroxides and separated. The provided cement solution contains calcium and magnesium ions. The constituents of Portland cement are: - CaO (60-67%), SiO₂ (17-25%), Al₂O₃ (3-8%), Fe₂O₃ (0.5-6%), MgO(0.1 -4%), SO₃(1-3%), K₂O & Na₂O (0.5-1.5%) and CaSO₄ (3-5%)

To estimate the calcium content in the given solution, a known volume of cement solution in presence of Magnesium, Calcium ions is titrated with standard EDTA solution using Patton & Reeder's indicator in the pH range 12-14. The indicator combines with Calcium ions to form a wine red colored Calcium-Indicator complex (Wine red). Near the end point, when free calcium ions are exhausted in the solution, further addition of EDTA, dissociates Calcium-Indicator complex, consumes the calcium ions and release free indicator which is blue in color. Therefore color change is wine red to blue.

Procedure:**Part A: Preparation of standard solution of Disodium salt of EDTA**

Weigh out the given EDTA crystals accurately into a 250 ml volumetric flask. Add quarter test tube of ammonia. Dissolve in distilled water and dilute up to the mark, mix well.

Part B: Estimation of CaO in the given cement solution

Pipette out 25 cm³ of the given Cement solution into a clean conical flask. Add 5 ml of glycerol, 5 ml of diethyl amine and 10 ml of 4N NaOH solution. Add 3-4 drops of Patton and Reeder's indicator. Titrate against standard EDTA solution till the color change from wine red to clear blue. Repeat the titration to get concordant values.

RESULT: CaO in the given sample of cement solution = -----

Observation:**Part A: Preparation of standard solution of Disodium salt of EDTA**Weight of weighing bottle + EDTA salt (W_1) = ----- gWeight of empty weighing bottle (W_2) = ----- gWeight of EDTA transferred ($W_1 - W_2$) = ----- g

$$\text{Molarity of EDTA} = \frac{\text{Weight of EDTA taken } (W_1 - W_2) \times 4}{\text{Molecular weight of EDTA (372)}} = \text{-----}$$

$$= \text{-----}$$

Part B: Estimation of CaO in the given cement solution

Burette reading	I	II	III
Final burette reading			
Initial burette reading			
Volume of EDTA run down (ml)			

Calculation:Volume of EDTA consumed by 25 cm³ of cement solution = -----

Weight of Cement in 25ml = 0.1g

1000 cm³ of 1 M EDTA = 56.08 g of CaO (Molecular mass of CaO = 56.08)

$$\text{----- cm}^3 \text{ of ----- M EDTA} = \frac{\text{X} \quad \text{X } 56.08}{1000 \times 1} \text{ g of CaO} = \text{-----}$$

$$= \text{-----}$$

25 cm³ of the Cement solution contains ----- g of CaO

$$\% \text{ of CaO in the given sample of cement solution} = \frac{\text{X } 100}{0.1} = \text{-----}$$

% of CaO in the given sample of cement solution = -----

Experiment No. 11**Date:** ___ / ___ / _____**DETERMINATION OF COD OF WASTE WATER****Aim:** To determine the COD of waste water***Principle:***

The chemical oxygen demand test is extensively employed as a means of measuring the pollution strength of industrial wastes. Chemical oxygen demand is a measure of the total quantity of oxygen required for complete 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 losses to sewer systems. Results may be obtained within a relatively short time and measures taken to correct errors on the day they occur.

Waste water contains organic impurities which include straight chain aliphatic compounds, aromatic hydrocarbons, straight chain alcohols, acids and other oxidisable materials. Straight chain compounds, acetic acid, etc., are oxidized 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 only partially by this procedure. This difficulty is overcome by adding mercuric sulphate to the sample.

Procedure:**Preparation of standard FAS solution (Mohr's salt solution)**

Weigh out the given FAS crystals accurately into a 250 ml volumetric flask. Add 2 test tube of H_2SO_4 . Dissolve in distilled water and dilute up to the mark, mix well.

Back titration:

Pipette out 25 ml of the waste water sample into a conical flask. Add 10 ml of standard potassium dichromate solution followed by 30ml of 6N sulphuric acid (containing silver sulphate) with constant shaking. Add boiling chips to the flask. Attach a reflux condenser and reflux the mixture for half an hour. Cool, wash the condenser with ion exchange water and transfer the washings to the flask. Add 2-3 drops of ferroin indicator and titrate against standard Mohr's salt solution until the solution turns from bluish green to reddish brown. Repeat for agreeing values.

Blank Titration:

Pipette out 10 ml of standard potassium dichromate solution. Add 30 ml of 6N Sulphuric acid (containing silver sulphate), reflux for half an hour. Cool and add 2-3 drops of ferroin indicator. Titrate against standard Mohr's salt solution until the colour turns from bluish green to reddish brown.

RESULT: The COD of given waste water sample contains ----- mg of O_2/dm^3

Observation:

Weight of weighing bottle + FAS crystals (W_1) = ----- g

Weight of empty weighing bottle (W_2) = ----- g

Weight of FAS crystals transferred ($W_1 - W_2$) = ----- g

$$\text{Normality of FAS} = \frac{(W_1 - W_2) \times 4}{\text{Equivalent weight of FAS (392)}} = \text{-----} \text{ N}$$

$$= \text{-----} \text{ N}$$

Burette reading	I	II	III
Final burette reading			
Initial burette reading			
Volume of FAS run down (ml)			

Calculation:

Volume of FAS consumed in Back titration = ----- cm^3 ($Y \text{ cm}^3$)

Volume of FAS consumed in Blank titration value = 50 cm^3 ($X \text{ cm}^3$)

Volume of FAS consumed for oxidizing organic mater ($X - Y$) = ----- cm^3

1000 cm^3 of 1 N FAS solution = 1 equivalent of oxygen = $8 \times 10^3 \text{ mg}$ of oxygen

$$(X - Y) \text{ cm}^3 \text{ of } N_{\text{FAS}} \text{ solution} = \frac{8 \times 10^3 \times (X - Y) \times N_{\text{FAS}}}{1000} = \text{-----}$$

$$= \frac{8 \times 10^3 \times X}{1000} = \text{-----}$$

= ----- mg of oxygen

25 cm^3 of waste water sample contains ----- mg of oxygen

$$1000 \text{ cm}^3 \text{ of waste water sample} = \frac{X \times 1000}{25} \text{ mg of oxygen} = \text{-----}$$

= ----- mg of oxygen/ dm^3

COD of waste water sample = ----- mg of oxygen / dm^3

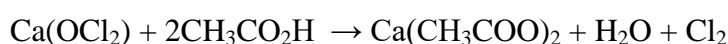
Experiment No. 12

Date: ___ / ___ / _____

ESTIMATION OF PERCENTAGE OF AVAILABLE CHLORINE IN THE GIVEN SAMPLE OF BLEACHING POWDER (IODOMETRIC METHOD).

Aim : Determine the percentage of available chlorine present in the given sample of bleaching powder.

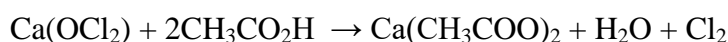
Theory: Bleaching powder, also known as chlorinated lime, is a yellowish-white powder having a smell of chlorine and is readily soluble in water. It is prepared by passing chlorine gas over slaked lime at a temperature of 35-450 and consists of a mixture of calcium hypochlorite $\text{Ca}(\text{OCl})_2$ and calcium chloride CaCl_2 ; in addition some amount of free slaked lime i.e. $\text{Ca}(\text{OH})_2 \cdot \text{H}_2\text{O}$ is generally present. Of these, $\text{Ca}(\text{OCl})_2$ is responsible for the bleaching action of bleaching powder. On treatment with glacial acetic acid, it liberates chlorine gas (Cl_2) as per the following reaction.



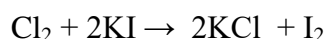
The amount of chlorine liberated by the action of an acid on bleaching powder (CaOCl_2) is termed as available chlorine. The chlorine content of bleaching powder varies from 35 – 40%. Besides bleaching action it has got strong germicidal and disinfectant properties also. Accordingly, it finds application as a disinfectant for drinking water or swimming pool water. Industrially, the bleaching powder finds major use in chemical, paper, textile and oil industries. The bleaching, oxidizing or disinfecting potential of a sample of bleaching powder depends on the percentage of chlorine liberated on action of acid. We may define available chlorine to be the grams of chlorine liberated from 100 g of the bleaching powder on treatment with dilute acid. Due to its hygroscopic nature, bleaching powder absorbs moisture from atmosphere and evolves chlorine. Due to this deterioration, a sample of bleaching powder may always contain lesser amount of chlorine than expected and therefore a sample of bleaching powder needs to be analyzed for its effective or available chlorine.

Principle :

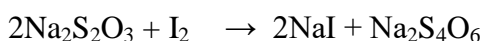
As mentioned in the introduction, a sample of bleaching powder liberates chlorine gas (Cl_2) on treatment with glacial acetic acid, as per the following reaction,



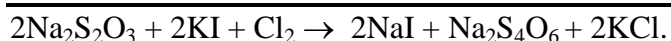
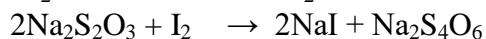
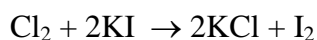
The amount of chlorine so liberated is termed as available chlorine. The liberated chlorine can be used to oxidize KI (taken in excess) in presence of acid and liberate out an equivalent amount of iodine as per the following equation:



This iodine can then be determined by titrating against a standardised solution of sodium thiosulphate using freshly prepared starch solution as an indicator. The chemical reactions involved can be given as follows,



The overall reaction between the chlorine liberated from the bleaching powder and sodium thiosulphate mediated by potassium iodide can be written as follows.



The sodium thiosulphate can be standardized by titrating against a primary standard solution of potassium dichromate.

Procedure : Dissolve 1g bleaching powder in 1 litre of distilled water. Pipette out 25 ml bleaching powder solution in to a clean conical flask, add 5 ml acetic acid and 10 ml KI solution. Titrate against 0.025 N sodium thiosulphate solution until a pale yellow colour is obtained. Add 1mL of starch solution and continue the titration until the blue colour disappears. Note down the volume of sodium thiosulphate solution as V_1 . Repeat the titration till concordant values obtained.

Blank titration : Pipette out 25 ml of distilled water in to a clean conical flask, add 5 ml acetic acid, 10 ml KI solution and 1ml of starch solution, if blue colors occurs titrate against 0.025 N sodium thiosulphate solution until blue colour disappears. Note down the volume of sodium thiosulphate solution as A_1 . If no blue colour occurs, titrate with 0.025 N iodine solution until a blue colour appears. Note down the volume of iodine as A_2 . Then, titrate with 0.025 N sodium thiosulphate solution till the blue colour disappears. Record the volume of sodium thiosulphate solution added as A_3 . Note down the difference between the volume of iodine solution and sodium thiosulphate as A_4 ($A_4 = A_2 - A_3$).

Observation

Bleaching powder solution x Standard sodium thiosulphate solution (0.025 N)

Burette reading	1	2	3
Final reading			
Initial reading			
Volume run down in cm^3 (V_1)			

Observation:**Part A:**

Mass of weighing bottle + Bleaching powder = W_1 = _____ g

Mass of weighing bottle after transferring the substance = W_2 = _____ g

Mass of bleaching powder transferred = $(W_1 - W_2)$ = _____ g

Part B:

Burette : $\text{Na}_2\text{S}_2\text{O}_3$ solution

Conical flask : 25 cm^3 of Bleaching powder suspension

Indicator : Starch (near the end point)

End Point : Dark blue to colourless.

Distilled water \times Standard sodium thiosulphate solution (0.025 N)

Burette reading	1	2	3
Final reading			
Initial reading			
Volume run down in cm^3			

Distilled water \times Standard iodine solution (0.025N)

Burette reading	1	2	3
Final reading			
Initial reading			
Volume run down in cm^3 (A_2)			

Calculation

$$\text{mg of Cl}_2/\text{ml} = \frac{(V_1 - A_1) \text{ or } (V + A_4) \times N \times 35.46}{\text{mL of bleaching powder solution taken}}$$

1000 mL of bleaching powder solution contains $1000 \times B$ mg of Cl_2
 i.e., 1000 mg bleaching powder contains $1000 B$ mg of Cl_2

Therefore, 100 mg of bleaching powder contains $\frac{1000 \times B}{10}$

% of Available chlorine =

Result:

Available chlorine in the given bleaching powder is. ... %

LIST OF EQUIPMENTS

Experiment: Estimation of FAS potentiometrically using standard $K_2Cr_2O_7$ solution.



POTENTIOMETER with built-in magnetic stirrer & standard cell

Readout	3 ½ digit LED display
Accuracy	.001 Volts
Power required	9V. dry cell or 230 V AC \pm 10% 50 Hz, 2 VA
Warming time	5 minutes
Weight	1 Kg (approx.)
Body	ABS
Dimension	95 mm (H) x 225 mm (W) x 215 mm (L)
Standard cell	1.018 V
Stirrer speed	200 RPM fixed
Accessories	Electrode clamp, Rod, Teflon magnet, Screwdriver and Dust proof cover

Experiment: Estimation of Copper colorimetrically.**The advantages of the Digital Photoelectric Colorimeter 112 at a glance:**

SPECIFICATIONS	112
Wavelength Range	400 nm to 700 nm with 8 optical filters
Filter's peak wavelength (typical)	420 nm, 440 nm, 490 nm, 520 nm, 540 nm, 570 nm, 600 nm, 700 nm.
Measuring Modes	%T & Abs
Photometric Resolution	1% T & 0.01 in Abs upto 1.99 Abs (O.D.)
Display	3-Digit LED
K-Factor Range	
Concentration	
Memory	
Sample Volume	1ml in 4ml Test tube
Source	White LED
Detector	Hermetically Sealed photodiode
Printer Port	
Power	230V \pm 10%, 50 Hz
Dimension	160 (W) X 200 (D) X 100 (H) mm
Weight	1.5 Kg (Approx)
Accessories	a) Four test tubes

Experiment: Estimation of Acids in acid mixture conductometrically.**CONDUCTIVITY METER with inbuilt magnetic stirrer & standard conductance**

Readout	3 ½ digit LED display
Unit of meas.	SIEMENS
Range	.01 μ S to 200 mS in six auto ranges
Six ranges	200 mS, 20mS, 2mS, 200 μ S, 20 μ S, 2 μ S
Accuracy	\pm 1% \pm last 2 digits
Power required	230 V AC 10%, 50 Hz, 6 VA
Weight	1 Kg. (Approx.)
Body	ABS
Warming period	5 minutes
Dimension	95 mm (H) x 225 mm (W) x 215 mm (L)
Standard cond.	Conductance of 1.000 mS
Electrode	Cell K = 1 - PVC sleeved
Stirrer speed	200 RPM Fixed
Accessories	Teflon magnet, Electrode clamp, Rod, Screwdriver, and Dust proof cover

Experiment: Determination of pKa of weak acid using pH meter

SPECIFICATIONS	335
pH Range	: 0 to 14.00 pH
Millivolt Range	: 0 to ± 1999 mV
Slope Correction	: 85% to 115%
Resolution	: 0.01 pH, 1 mV in mV mode
Repeatability	: ± 0.01 pH ± 1 digit for pH ± 1 mV ± 1 digit for mV
Standardization Range	: ± 1 pH (Approx)
Temp. Compensation	: 0 to 100° C Dig. Display (Manual)
Display	: 3½-Digit Red LED Seven Segment with automatic polarity & decimal point
Polarization Current	:
Recorder Output	:
Power	: 230V $\pm 10\%$ 50 Hz
Dimension	: 235 (W) X 185 (D) X 85 (H) mm
Weight	: 1.25 Kg (Approx)
Accessories	: Combined Electrode Stand & Clamp
Optional Accessories	: Pt. Electrode & Calomel Electrode for Potentiometric Titration Temp. Sensor pt 100

DETERMINATION OF p^{K_a} OF A WEAK ACID USING pH METER

- How is p^{K_a} of a weak acid evaluated from the graph?
pH at half equivalence point gives the pK_a of a weak acid
- What is a weak acid?
Weak acids are weak electrolytes, which ionize incompletely in aqueous solution.
- Give an example for weak acid?
Acetic acid, Oxalic acid
- What is p^{K_a} of a weak acid?
 pK_a is defined as, negative logarithm to the base 10 of dissociation constant of weak acid. i.e.,
 $pK_a = -\log_{10}K_a$, Where K_a is the dissociation constant of the weak acid.
- What is meant by p^H ?
pH is defined as negative logarithm to base 10 of hydrogen ion concentration. $p^H = -\log_{10}[H^+]$
- What is the modern definition (IUPAC) of p^H ?
The difference in pH between two solutions S (std) and X (unknown) at the same temperature with the same reference electrode and with hydrogen electrodes at the same hydrogen pressure is given by

$$pH(X) - pH(S) = \frac{E_X - E_S}{2.3026 RT / F}$$
- Why glass electrode is called an ion selective electrode?
Because it is able to respond to certain specific ions (H^+ ions) only and develop a potential while ignoring the other ions in a solution.
- How is the measurement of p^H made?
The measurement of pH is made by determining the emf of the cell containing glass electrode and the calomel electrode immersed in the test solution. The emf of the cell is expressed by the equation $E = K + 0.0591 pH$, where K is a constant.
- How are p^H and p^{K_a} related?
According to Henderson-Hasselbalch equation, $p^H = p^{K_a} + \log_{10} [\text{salt}] / [\text{acid}]$
- Why pH at half equivalence point is equal to p^{K_a} ?
At half equivalence point concentration of salt = concentration of acid, Therefore $p^H = p^{K_a}$.
- How are p^{K_a} and strength of a weak acid related?
Higher the pK_a , lower will be the strength of the weak acid.
- What are the electrodes used in the measurement of pH and determination of p^{K_a} ?
Glass electrode (indicator electrode) and calomel electrode (reference electrode).
In this case, glass electrode acts as anode & calomel acts as cathode.
- Why is pH increases suddenly after the equivalence point?

At the equivalence point, all the weak acid has been neutralized by the base. Afterwards the concentration of hydroxyl ions increases. So pH increases suddenly.

POTENTIOMETRIC TITRATION-ESTIMATION OF FAS USING STANDARD POTASSIUM DICHROMATE SOLUTION

1. What is single electrode potential?

The potential that is developed when an element is in contact with a solution containing its own ions is called single electrode potential

2. What is standard electrode potential?

The potential that is developed when an element is in contact with a solution containing its own ions in 1M concentration at 298 K is called standard electrode potential

3. What is meant by emf?

EMF is potential difference required to drive current across the electrodes.

$$EMF = E_{\text{cathode}} - E_{\text{anode}}$$

4. What is potentiometer?

It is a device or circuit used for measuring the emf between the two electrodes

5. What is potentiometric titration?

It is the measurement of emf to determine the concentration of ionic species in solution is called potentiometry

6. What is the principle of potentiometric titration?

The measurement of the emf between two electrodes (indicator and reference) and to determine the equivalence point of redox reaction

7. Which are the electrodes used in potentiometric electrode?

Platinum electrode as indicator electrode (anode) and calomel electrode as reference electrode (cathode)

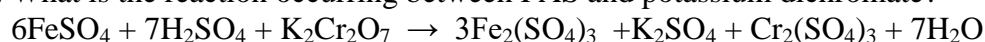
8. What is determining factor in the oxidation-reduction reaction?

The determining factor is the ratio of the concentrations of the oxidized and reduced forms.

9. What is indicator electrode?

The indicator electrode is an electrode whose potential is dependent upon the concentration of the ion to be determined.

10. What is the reaction occurring between FAS and potassium dichromate?



Acidified potassium dichromate oxidizes ferrous sulphate to ferric sulphate and itself gets reduced to chromic sulphate

11. Why sulphuric acid is added to FAS solution during emf measurement?

In presence of sulphuric acid, potassium dichromate oxidizes ferrous ion (Fe^{+2}) to ferric ion (Fe^{+3})

12. Why the colour of the solution changes to green on adding potassium dichromate?

Due to formation of green coloured chromic sulphate.

13. Why emf rapidly change at the equivalence point?

At the equivalence point, emf depends on concentration of both ferric ion and

dichromate ion .Therefore emf rapidly change at the equivalence point.

14. What are the advantages of potentiometric titrations?
 - i) Turbid, fluorescent, opaque or coloured solutions can be titrated
 - ii) Mixture of solutions or very dilute solutions can be titrated.
 - iii) The results are more accurate

COLORIMETRIC DETERMINATION OF COPPER

1. What is the basic principle of colorimetric measurements?

It consists of comparing under well defined conditions, the colour produced by the substances in amount with the same colour produced by a known amount of material being determined.
2. What is colorimetry?

Chemical analysis through measurements of absorption of light radiation in the visible region of the spectrum (400-760nm) with respect to a known concentration of the substance is known as colorimetry
3. What forms the basis for colorimetric determination?

The variation of colour of a system with change in concentration of some component forms the basis for the colorimetric determination
4. What is photoelectric colorimeter?

It is an electrical instrument, which measures the amount of light absorbed using a photocell.
5. What are filters? Why are they used?

The filter consists of either thin film of gelatin containing different dyes or of coloured glass. The filters are used in colorimeter for selecting any desired spectral region.
6. What is wavelength?

The distance between any two successive peaks or troughs of waves is called wave length. It is represented by λ
7. What is wave number?

It is reciprocal of wavelength. $\text{Wave number} = 1/\lambda = \text{Frequency} / \text{Velocity of light}$
8. What is frequency?

It is the number of waves passing through a point per second. It is represented by ν
9. State Beer's law.

The intensity of a beam of monochromatic light decrease exponentially as the concentration of the absorbing substance increases arithmetically.
10. State Lambert's law.

When a monochromatic light passes through a transparent medium, the rate of decrease in intensity with the thickness of the medium is proportional to the intensity of the light. or The intensity of the emitted light decreases exponentially as the thickness of the absorbing medium increases arithmetically.
11. State Beer-Lambert's law.

The amount of light absorbed is directly proportional to the concentration of the solution.

$A = \log I_0 / I_t = \epsilon cl$;(ϵ = Molar extinction coefficient, c = concentration, l = path length)
Therefore, $A \propto C$

12. What is calibration curve?

It is the plot of absorbance or Optical Density against concentration of solutions. For solutions obeying Beer's law, this is a straight line.

13. What is meant by transmittance?

It is the ratio of amount of light transmitted (I_t) to the total amount of light absorbed (I_0)

$$T = I_t / I_0$$

$$\text{Absorbance} = 1 / T = I_0 / I_t$$

14. What is absorbance or optical density?

Reciprocal of transmittance, I_0/I_t is called absorbance or optical density

$$A = \frac{\log I_0}{I_t} = \log 1/T = -\log T$$

15. How is optical density is related to the concentration of the substance?

The optical density is directly proportional to the concentration of the substance

16. What is a stock solution?

A stock solution is a standard solution in which a known amount of solute dissolved in a known volume of solvent.

17. Can different nessler's tubes be used during OD measurements of solutions?

No, because absorbance depends on the path length of the solution.

18. Mention a few advantages of photoelectric colourimetric determinations.

i) A colourimetric method will often give more accurate results at low concentrations than the corresponding titrimetric or gravimetric procedure.

ii) A colorimetric method may frequently be applied where no satisfactory gravimetric or titrimetric procedure exists i.e. for certain biological substance.

19. Why are different volumes of solution taken in the flask?

Different volumes of solutions are taken to prepare standard solutions of different concentration, which are used to plot a calibration curve.

20. What is blank solution?

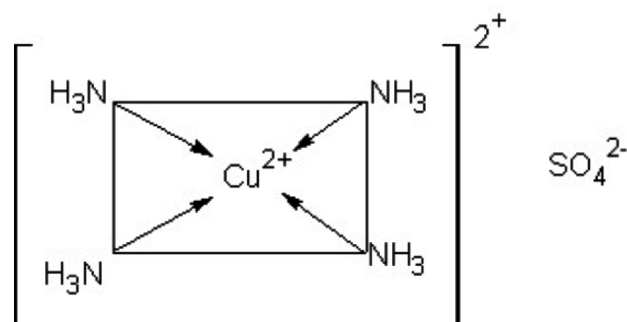
A blank solution is identical in all respect to the test solution except for the absence of test solute.

21. Why is a blank solution used in colorimetric estimation?

To nullify the absorbance caused due to the colouring impurities present in the reagents.

22. Name the complex obtained when ammonia is treated with copper sulphate solution

Cuprammonium sulphate, $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4$ a dark blue colour complex



23. Why is ammonia added? Why is that same amount of ammonia added?
Ammonia is added to get cuprammonium sulphate $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4$ a dark blue complex. Same amount of ammonia is added to nullify the absorbance due to any colouring impurities present in ammonia.
24. Why is estimation of copper done at 620 nm wave length?
The estimation of copper is carried at 620 nm wave length because the complex shows maximum absorbance at 620 nm.
25. Mention a few important criteria for a satisfactory colorimetric analysis.
- In order to obey Beer-Lambert's law, the solute must not undergo salvation, association, dissociation, hydrolysis or polymerization in the solvent used.
 - The colour produced should be sufficiently stable to permit an accurate reading to be taken.
 - Clear solutions free from traces of precipitate or foreign substances in either blank or standard test solution should be used.

CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE USING STD. NaOH SOLUTION.

- What is conductivity of an electrolyte solution?
Is a measure of its ability to conduct electricity?
- What is conductance?
Reciprocal of resistance is called conductance.
- What is a cell?
A device, which produces an EMF and delivers electric current as the results of a chemical reaction.
- What factors determine the conductance of a solution ?
Two factors determining the conductance of a solution are;
 - Mobility of ions (Higher the mobility, higher is the conductance) and
 - Number of ions (More the number of ions, more is the conductance).
- What is cell constant?
Is a function of the electrode areas, the distance between the electrodes and the electrical field pattern between the electrodes. Cell constant is defined as the ratio of the distance between the electrodes, d , to the electrode area, A .
- What is the unit of cell constant?
Commonly, expressed in cm^{-1} ,

7. State Ohm's law.
Current, I (ampere), flowing in a conductor is directly proportional to the applied electromotive force, E (volt) and inversely proportional to the resistance R (ohm) of the conductor. $I = E/R$.
8. What is the unit of conductance?
 $\text{Ohm}^{-1}\text{m}^{-1}$ or Sm^{-1} (S- siemens).
9. Mentions the different types of conductance.
 - i) Specific conductance.
 - ii) Equivalent conductance
 - iii) Molar conductivity.
10. Which of the above conductivity is measured during the conductometric titration ?
Specific conductance.
11. What is specific conductivity?
Conductivity of a solution between 1cm^2 area and kept 1 cm apart.
12. What is equivalent conductance?
Conductivity of a solution, which contains 1g equivalent of solute, when placed between 2 electrodes of 1cm apart. $\lambda = kV$.
13. What is molar conductivity?
Conductivity of the solution, which contains 1g molecular weight of a substance, when placed between two electrodes of 1 cm apart, $\mu = kV$.
14. What is the principle involved in conductometric titration?
There is a sudden change in conductance of a solution near the end point. Hence the end point is determined graphically by plotting conductance against the titer values.
Measuring resistance and the dimension of conductivity cell (Ohm law) and plotting the titration curve (conductance-as reciprocal of resistance against the volume of titrant) is the principle.
15. Explain the variation in conductivity at the end point.
In the case of NaOH and HCl experiment, addition of NaOH to HCl decreases the conductance because of the replacement of high conducting mobile H^+ ions (Conductivity : $350\text{ Ohm}^{-1}\text{m}^{-1}$) by the less mobile Na^+ ions (Conductivity : $50\text{ Ohm}^{-1}\text{m}^{-1}$). Trend continues till all the hydrogen ions are replaced and end point is reached. Further addition of NaOH, increases the conductance because of the presence of free OH^- ions (Conductivity : $198\text{ Ohm}^{-1}\text{m}^{-1}$).
16. Why are the slopes of curves before and after neutralization point not exactly the same ?
Before the neutralization point, the conductivity is mainly due to the highly mobile H^+ ions and after the end point, it is mainly due to OH^- ions, which are less mobile compared to H^+ ions. As a result, magnitude of decrease in conductivity is more before the neutralization point compared to that after the neutralization point.
17. How is the accuracy of the method determined?
Accuracy of the method is greater when the angle of intersection is more acute and more nearly the end points of the graph on a straight line.

18. What are the advantages of conductometric titration over visual potentiometric titrations?
- Accurate for both dilute and concentrated solutions.
 - Can be employed for colored solutions.
 - Very weak acids such as boric, phenol (cannot be titrated potentiometrically) can be titrated.
 - Mixture of acids can be titrated more accurately.

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

1. What is viscosity?

Viscosity arises due to internal friction between moving layers of molecules. A moving layer exerts a drag or friction on its nearest moving layer backward. This property of a liquid by which it retards or opposes motion between the layer is called viscosity.

2. What is viscosity co-efficient of a liquid?

The viscosity coefficient of a liquid 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.

3. What is density of liquid?

The density of a liquid is the mass of its unit volume.

4. The density of a substance is expressed relative to what?

The density of a substance is expressed relative to that of water at 4⁰C

5. What is specific gravity?

Specific gravity or relative density is the weight of a given liquid divided by the weight of an equal volume of water at the same temperature.

6. How are specific gravity and density of liquid related?

Density of liquid = specific gravity of the liquid x Density of water at the same temperature

7. What is the SI unit of viscosity coefficient?

Pascal second ($1 \text{ ps} = 1 \text{ kg m}^{-1} \text{ s}^{-1}$)

8. What is the unit of viscosity coefficient in cgs system?

Millipoise.

9. What are the factors that affect the viscosity of a liquid?

- Increase in molecular weight results in an increase in viscosity.
- Branched chain compounds have higher viscosity than those involving straight chain compounds.
- The polar compounds are more viscous than the non polar ones. The presence of hydrogen bonds cause the viscosity to increase.
- The viscosity of liquids increases by the presence of lyophilic colloids and suspended impurities.
- Temperature has marked influence on the viscosity of a liquid (about 2% per degree).

10. What is the law based on the viscous flow of liquids through capillary tubes?

Poiseuille law

$$\eta = \frac{\Pi \rho r^4 t}{8Vl}$$

Where $P = hgd$

V = volume of the liquid, r = radius of the tube, l = length of the tube,

ρ = pressure difference between two ends of the tube,

η = the coefficient of viscosity of the liquid,

t = time required for the volume.

11. How does viscosity vary with temperature?

The viscosity of a liquid usually decreases with rise in temperature.

12. Why should the viscometer be dried before the measurements are done?

The viscometer should be dried to avoid the formation of emulsion, which changes the rate of flow of the liquid.

13. Why is acetone used for cleaning viscometer?

Acetone is volatile organic liquid. To dry the viscometer quickly, it is rinsed with acetone.

14. Why is viscometer not rinsed with the given liquid or water?

If the viscometer is rinsed with given liquid or water before measuring the flow time, the volume taken will be more than a definite known volume.

15. Why do you require laboratory temperature for viscosity determination?

Because the physical constants like density and viscosity of a liquid vary with temperature.

16. How is the viscosity of a liquid related to its mobility?

Viscosity of a liquid is inversely proportional to its mobility.

17. What is fluidity of a liquid?

Fluidity of liquid is the reciprocal of viscosity co-efficient. It is a measure of the ease with which the liquid flows.

Fluidity (Θ) = $1/\eta$, η = the viscosity co-efficient.

FLAME PHOTOMETRIC ESTIMATION OF SODIUM IN THE GIVEN SAMPLE OF WATER.

1. What is flame photometry?
Flame photometry is an atomic emission method for the detection of metal salts like Sodium, Potassium etc, by measuring the flame emission of respective metal salt solutions.
2. What are alkali and alkaline earth metals?
The elements of first groups, are called alkali metals (Li, Na, K, Rb and Cs). Elements of second group are called alkaline earth metals (Be, Mg, Ca, Sr and Ba).
3. What are the various events that occur, when a solution containing metal atom ion Atomized through a flame?
Sequence of events taking place are ;
 - a. Aspiration of liquid sample into a flame.
 - b. Formation and evaporation of liquid droplets resulting in the formation of residue.
 - c. Decomposition of residue into neutral atoms.
 - d. Excitation of atoms and emission of radiation from atoms.
 - e. Measurement of wavelength and intensity of emitted radiation by flame photometer.
4. What are different components of a flame photometer ?

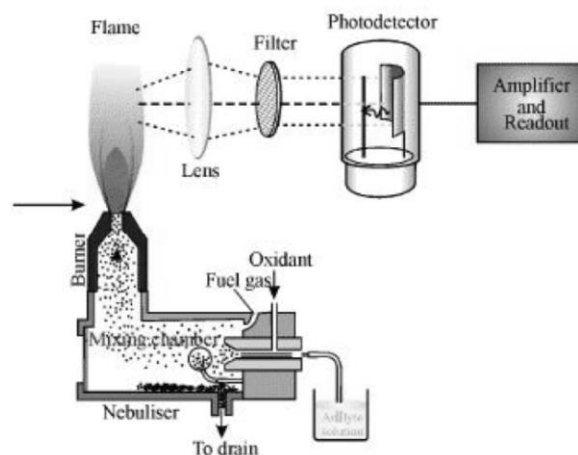


Fig. : Schematic diagram showing the layout of various components of a flame photometer

An atomizer, the burner system and an optical system, a photocell, amplifier and recorder.

5. Why the analysis of Sodium and Potassium is advantageous in flame photometry?
 1. Is fast, simple and reliable.
 2. Sensitivity in the ppm and sub-ppm range.
 3. Flame photometer is portable and its ease of operation.
6. What is the role of filter in flame photometry?
Permits only the radiation characteristics of the element under investigation.
7. What are the errors that occur in flame photometry?
An extra molecular entity formed lowers the efficiency. The low temperature renders this method susceptible to certain disadvantages, most of them related to interference and the stability (or lack thereof) of the flame and aspiration conditions.

8. What are the factors that influence this experiment?
Fuel, oxidant flow rates, purity aspiration rates, solution viscosity, contaminants in the samples etc affect the experiment.
9. Name the fuel and oxidant used in this experiment?
LPG-fuel and air as oxidant.
10. Why blank test is essential?
A solution of blank is run for setting zero emission.

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

1. What is Brass?

Brass is an alloy

2. What is an alloy?

An alloy is a homogeneous mixture of two or more metals

3. What are the constituents of brass?

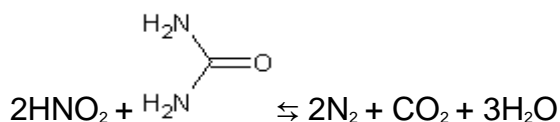
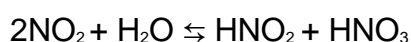
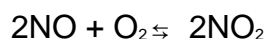
Brass is an alloy of copper (50-90%), zinc (20-40%), small quantities of tin, lead and iron.

4. How is brass solution prepared?

Brass solution is prepared by dissolving the brass foil in 1:1 nitric acid.

5. What is the purpose of adding urea to the brass solution?

Urea is added to destroy excess of nitrous acid and oxides of nitrogen which interfere with the determination. If they are not destroyed they also oxidize KI to I₂ which should have been done by Cu⁺² ions



6. Why ammonium hydroxide is added to the brass solution?

Ammonium hydroxide is added to neutralize the mineral acid (HNO₃ & HNO₂)

7. What is the need to neutralize the mineral acid?

The mineral acid HNO₃ is a strong oxidizing agent. It oxidize KI to I₂ therefore the amount of iodine liberated does not corresponds to the exact concentration of Cu²⁺ ions.

8. What is the bluish white precipitate formed after adding ammonia solution?

Cupric hydroxide

9. Why acetic acid is added?

Acetic acid is added to neutralize the excess of ammonium hydroxide and to make the solution slightly acidic.

10. Why is acidic medium necessary?

The oxidation of KI to I₂ takes place in acidic medium.

11. Why is KI added to brass solution although copper in brass is determined?

Cupric ions do not react with sodium thiosulphate solution. Cupric ions oxidize KI and iodine is liberated. The amount of iodine is liberated is equal to the amount of cupric ions present in the solution.

12. Although copper ions are blue in colour in the beginning become colorless after the end Point? Why?

At the beginning of the titration Cu²⁺ ions present (blue) which are reduced to Cu⁺ ions (colorless) state.

13. Why do you get blue color when starch indicator is added?

Triiodide in the solution reacts with the amylose of starch gives a blue color.

14. How is liberated iodine estimated?

The amount of iodine liberated is estimated by titrating it against standard sodium thiosulphate solution using starch as an indicator.

15. What is the reaction that occurs between iodine & sodium thiosulphate?**16. Why is starch indicator added towards end point?**

If the intensity of iodine is more, the starch indicator forms a stable water insoluble complex with iodine. As a result the volume of sodium thiosulphate will be more than expected.

17. What is the white precipitate produced at the end point?

Cuprous iodide

PREPARATION OF STANDARD POTASSIUM DICHROMATE SOLUTION AND DETERMINATION OF IRON IN THE GIVEN SAMPLE OF HAEMATITE ORE SOLUTION. (External indicator method)

1. What is an ore?

An ore is mineral, from which a metal can be extracted economically & conveniently

2. What is a mineral?

A mineral is a naturally occurring metallic compound.

3. What are the main constituents of haematite ore?

Ferric oxide (Fe₂O₃)

4. Give the other forms of iron ore?

Magnetite (Fe₃O₄), Siderite (FeCO₃), Iron pyrite (FeS₂)

5. Why HCl is added?

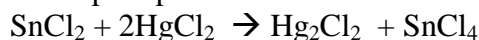
HCl is added to convert Fe₂O₃ to FeCl₃, as Fe₂O₃ does not react with K₂Cr₂O₇.

6. What is the role of stannous chloride?

Stannous chloride reduces Ferric ion to ferrous ion and a slight excess is added to ensure complete reduction. $2\text{FeCl}_3 + \text{SnCl}_2 \rightarrow 2\text{FeCl}_2 + \text{SnCl}_4$

7. Why is mercuric chloride added?

To remove excess of stannous chloride. Mercuric chloride reacts with stannous chloride to form a silky white precipitate of mercurous chloride.(Calomel)

**8. What happen when excess of stannous chloride is not removed?**

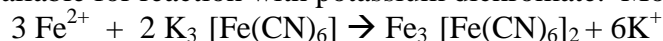
The ferric ions formed during the course of the titration get reduced to ferrous ions. As a result, the volume of potassium dichromate consumed will be more.

9. Which is the indicator used?

$\text{K}_3[\text{Fe}(\text{CN})_6]$, Potassium ferri cyanide as external indicator.

10. Why potassium ferri cyanide cannot be used as an internal indicator?

Because it combines irreversibly with ferrous ion to form a deep blue ferrous ferri cyanide complex (Turnbull's blue). These ferrous ions involved in complex formation are not available for reaction with potassium dichromate. Moreover end point cannot be detected.

**11. Why potassium Ferro cyanide cannot be used as an indicator in the estimation of Fe**

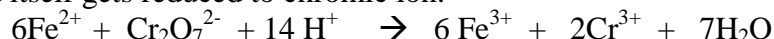
Potassium Ferro cyanide cannot be used as an indicator because Ferro cyanide does not react with ferrous ion.

12. Why the colour of the indicator drop remains same at the end point?

At the end point there are no more ferrous ions available to react with the indicator, as they oxidized to ferric ions by the time the end point is reached.

13. What is the reaction that occurs during the titration?

Acified potassium dichromate oxidizes ferrous iron present in the haematite solution to ferric iron and itself gets reduced to chromic ion.

**14. Can Fe be determined using internal indicators?**

Yes, Fe can be determined using internal indicators.

15. Name the internal indicator used for iron determination?

Ex: a) Diphenyl amine b) N-phenylanthranilic acid

PREPARATION OF STANDARD EDTA SOLUTION AND DETERMINATION OF TOTAL HARDNESS OF A SAMPLE OF WATER**1. Why is ammonia ammonium chloride buffer added?**

Ammonia ammonium chloride is added to maintain a pH of 10. Otherwise pH decreases as H^+ ions are released due to the substitution of metal ions for H^+ ions in EDTA.

2. What is hard water?

Hard waters are a kind of water that requires considerable amounts of soap to produce foam and lather.

3. What is total hardness of water?

The sum of both temporary and permanent hardness is called total hardness of water.

4. How are the waters classified based on the degree of hardness?

Soft water	0 to 75 mg / litre
Moderately hard water	75 to 150 mg / litre
Hard water	150 to 300 mg / litre
Very hard water	> 300 mg / litre

5. How is hardness of water caused?

Hardness of water caused by divalent metallic cations in association with anions such as HCO_3^- , SO_4^{2-} , Cl^- , SiO_3^{2-} etc. the principal hardness causing cations are calcium and magnesium.

6. What is the difference between temporary and permanent hardness?

Temporary hardness is due to unstable bicarbonates of Ca and Mg, while permanent hardness is due to more stable Cl^- and SO_4^{2-} of Ca and Mg

7. How temporary hardness is be removed?

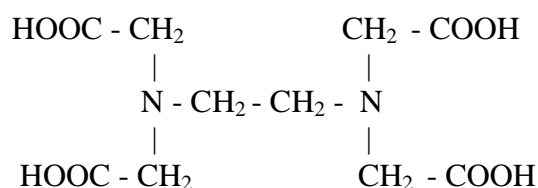
The temporary hardness of water can be removed by boiling water during which bicarbonates decomposes to give carbonates.

8. How do you express the total hardness of water?

It is expressed as parts per million of CaCO_3 .

9. What is EDTA?

Ethylene diamine tetra acetic acid.

10. Write a structural formula of EDTA.**11. How many replaceable hydrogen atoms are present in disodium salt of EDTA?**

Two.

12. Why is disodium salt of EDTA preferred to EDTA?

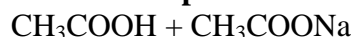
Because EDTA is sparingly soluble in water.

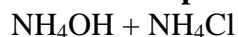
13. Why is ammonia solution added while preparing EDTA solution?

Because to increase the rate of dissolution of EDTA salt.

14. What is buffer capacity?

The capacity of a solution to resist the change in pH

15. Give an example of acidic buffer?

16. Give an example of basic buffer?**17. What is reserve alkalinity?**

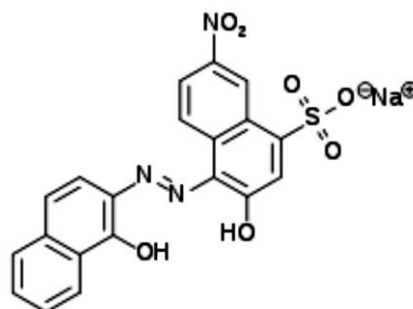
Buffer solutions are considered to possess reserve acidity as well as alkalinity. According to Henderson-Hasselbalch equation, $\text{pH} = \text{pK}_b + \log_{10} [\text{salt}] / [\text{base}]$, At half equivalence point [Concentration of salt = Concentration of base], Therefore $\text{pH} = \text{pK}_b$. Similarly, for reserve acidity for acidic buffer.

18. What is buffer solution?

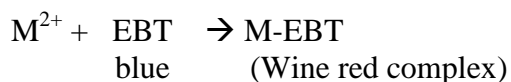
The solution which resists change in its pH value even after adding small amounts of an acid or base to it.

19. What is the chemical name of EBT ?

Sodium 4-[(1-hydroxynaphthalen-2-yl)hydrazinylidene]-7-nitro-3-oxo-*Y*-naphthalene-1-sulfonate.

**20. Why is EBT shows wine red at the beginning and blue colour at the end?**

EBT is blue in colour, which is added to hard water with a pH of 10; it combines with a few of the calcium and magnesium ions to form a weak complex which is wine red in colour as shown in the equation.



This action frees the EBT from the complex (M-EBT), and the wine red color changes to a blue colour at the end of titration.

21. Titrations involving EDTA carried out slowly towards the end point. Justify?

Because the rate of formation of the metal complex of EDTA is very slow.

22. What is the application of hardness data in environmental engineering practice?

- i) Hardness of water is an important consideration in determining the suitability of a water for domestic and industrial uses.
- ii) Determination of hardness serves as a basis for routine control of softening processes.

PREPARATION OF STANDARD EDTA SOLUTION AND DETERMINATION OF PERCENTAGE OF CALCIUM OXIDE IN THE GIVEN SAMPLE OF CEMENT SOLUTION. (Rapid EDTA Method)

1. What is cement?

Cement is a lime based building material used to bind together coarse aggregates.

2. Mention the composition of Portland cement.

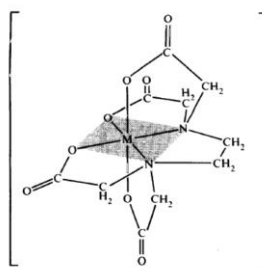
CaO = 60-66%, SiO₂ = 17-25%, Al₂O₃ = 3-8%, Fe₂O₃ = 2-6%, MgO = 0.1 – 5.5%, Na₂O & K₂O = 0.5 – 1.5% and SO₃ = 1-3%

3. How is cement solution prepared?

Accurately weighed amount of cement is warmed with moderately conc. HCL till cement dissolves insoluble silica is filtered off & filtrate is the cement solution

4. Why is the titration called rapid EDTA method?

The titration is called rapid EDTA method because calcium ions in cement solution are estimated directly & quickly by using standard EDTA solution without removing the other metal ions.



5. What is the role of NaOH in this titration?

NaOH reacts with magnesium ion & precipitates as magnesium hydroxide from the cement solution. $Mg^{2+} + 2NaOH \rightarrow Mg(OH)_2 + 2Na^+$

6. What are the constituents of cement?

Oxides of calcium, magnesium, iron, aluminum and silicon.

7. What is the main constituent of cement?

Calcium oxide.

8. What is the role of glycerol?

Glycerol is added to get sharp end point.

9. What is the role of NaOH?

NaOH is added to mask the magnesium ions.

10. What is the function of diethyleamine?

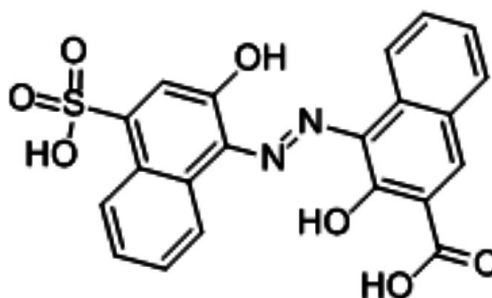
Diethyleamine is added to maintain a pH of about 12.5.

11. Why is EBT indicator cannot be used in this experiment?

Because it forms a weak complex with calcium ions at pH range of 12-14.

12. Which is the indicator used in the determination of CaO in cement solution?

Patton and reeder's reagent (Calconcarboxylic acid; IUPAC name : Naphthalenecarboxylic acid, 3-hydroxy-4-[(2-hydroxy-4-sulfo-1-naphthalenyl)azo]-; 3-hydroxy-4-[(2-hydroxy-4-sulfo-1-naphthalenyl)azo]-2-Naphthalenecarboxylic acid).



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

1. What is sewage?

Sewage is commonly a cloudy dilute aqueous solution containing human & household waste, industrial waste, ground waste, street washings. Sewage contains organic and inorganic matters in dissolved, suspended & colloidal states.

2. What is meant by industrial sewage?

The waste water coming out of industries such as chemical plant, fertilizer industries, leather tanneries, sugar and paper industries, breweries, textile mills, oil refineries, pharmaceutical units is called industrial sewage.

3. What is COD?

It is amount of oxygen required for the complete chemical oxidation of both organic and inorganic matter present in the sample of water by a strong chemical oxidizing agent such as acidified potassium dichromate.

4. What are the compounds present in industrial waste water sample?

Waste water contains organic impurities which include straight chain aliphatic compounds aromatic hydrocarbons, straight chain alcohols, acids and other oxidisable materials.

5. What is BOD?

It is the amount of oxygen required for the complete oxidation of organic matter present in liter of waste water over period of 5 days at 20^o C.

6. Why COD greater than BOD value?

Because in COD both organic & inorganic matters gets oxidized & in BOD only organic matters gets oxidized.

7. What are end products of oxidation?

CO₂ & H₂O.

8. Why H₂SO₄ is added to conical flask?

Because Potassium dichromate acts as an oxidizing agent in presence of H₂SO₄.

9. What is the unit of COD?

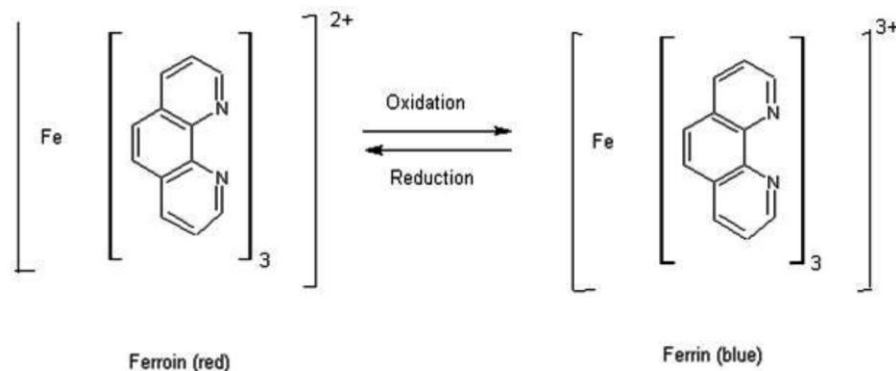
mg/litr of O₂ or mg/dm³ of O₂ or ppm of O₂ .

10. Name the oxidizing agent used in the COD test?

Potassium di chromate.

11. Explain the colour change during the titration.

Ferriin indicator is red in colour in the reduced form with the composition, $[\text{Fe}(\text{O-Phen})_3]^{2+}$ (ferriin), When ferriin is added to conical flask containing waste water & potassium dichromate. The indicator gets oxidized by potassium dichromate (oxidizing agent) to form $[\text{Fe}(\text{O-Phen})_3]^{3+}$ (ferrin), which is bluish green. This solution is titrated with FAS taken in the burette (reducing agent) which reduces ferrin to Ferriin which is in red colour which is the end point of titration.

**Active ingredient ions****12. What is back titration?**

Back titration is a technique used to find the concentration of the reactant by reacting with an excess volume of reactant of known concentration.

13. Differentiate between blank & back titration. Why blank value is higher than back value?

Blank titration is done using distilled water instead of test solution (waste water) and back titration is done using waste water as one of the solvent. Blank titration involves no oxidation of organic matter.

14. What general groups of organic compounds are not oxidized in the COD test?

Aromatic hydrocarbons and pyridine are not oxidized in COD test.

15. What is the role of silver sulphate?

Silver sulphate acts as a catalyst in the oxidation of straight chain aliphatic hydrocarbons and acetic acid. Oxidation is effective in presence of silver ions.

16. What is the role of mercuric sulphate?

Chloride ions normally present in high concentration in waste water undergo oxidation in COD test and cause erroneously high results. Mercuric ions of mercuric sulphate bind the halide ions present in waste water to form poorly ionized mercuric chloride and prevent the precipitation of silver halide by making halide ions unavailable.

17. If nitrites are present as impurities, what is the reagent used?

Sulphamic acid (amido sulphonic acid, H_3NSO_3)

18. What are the products formed after COD analysis?

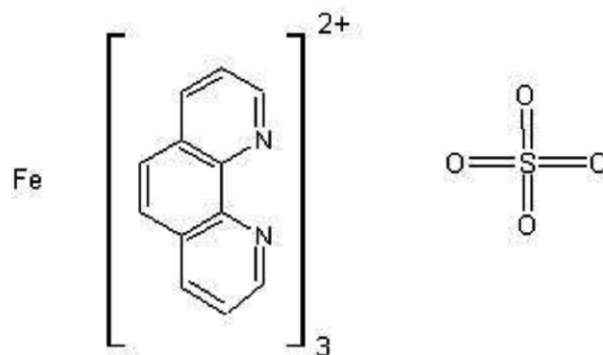
During COD analysis organic matter is completely oxidized to carbon dioxide and water by acidified potassium dichromate solution.

19. Why is sulphuric acid added during the preparation of std. FAS solution?

Sulphuric acid is added to prevent the hydrolysis of ferrous sulphate into ferrous hydroxide.

20. What is the composition of ferroin?

Ferroin is ferrous 1, 10-Phenanthroline sulphate.



Ferroin indicator

21. Why blank titration is necessary?

Because the COD measures the oxygen demand in the sample of water, it is important that no outside organic material be accidentally added to the sample to be measured. To control for this, so called blank sample is required.

22. Mention a few applications of COD test in environmental engg.?

- i) The COD test is extensively used in the analysis of industrial wastes
- ii) It is particularly valuable in survey designed to determine and control losses to sewer systems
- iii) The COD is helpful in indicating toxic conditions and the presence of biologically resistant organic substances.

23. What is the limitation of COD?

One of the chief limitations of COD test is inability to differentiate between biologically oxidisable and biologically inert organic matter. Also, it does not provide any evidence of the rate at which the biologically active material would be stabilized under conditions that exists in nature.

DETERMINATION OF AVAILABLE CHLORINE IN BLEACHING POWDER

1. What is meant by 'available chlorine'?

The amount of chlorine liberated by the action of dilute mineral acids on bleaching powder.

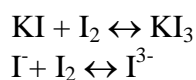
2. What is meant by iodometric titration?

Titration in which liberated iodine is titrated against standard solution of sodium thiosulphate is called iodometric titration.

3. What is the formula of bleaching powder?

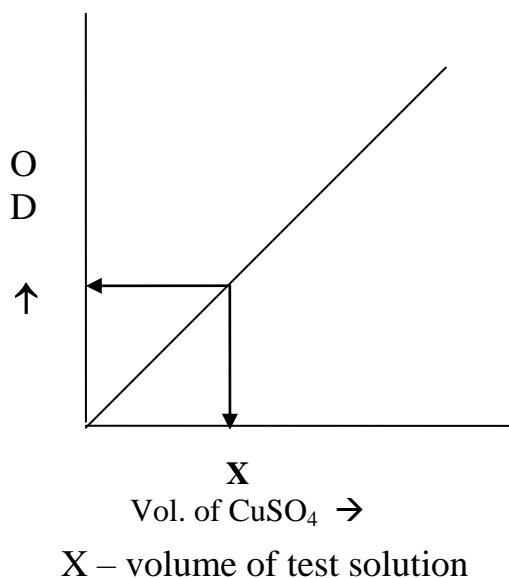
CaOCl_2

4. Write the chemical reaction taking place in this reaction.
 $\text{CaOCl}_2 + 2\text{CH}_3\text{CO}_2\text{H} \rightarrow (\text{CH}_3\text{CO}_2)_2\text{Ca} + \text{H}_2\text{O} + \text{Cl}_2 \uparrow$
 $\text{KI} + \text{Cl}_2 \rightarrow 2\text{KCl} + \text{I}_2$
 $2\text{Na}_2\text{S}_2\text{O}_3 + \text{I}_2 \rightarrow \text{Na}_2\text{S}_4\text{O}_6 + 2\text{NaI}$
5. What is the other name of bleaching powder?
 Chloride of lime or Bleach.
6. What is approximate percentage of available chlorine in fresh commercial bleaching powder?
 36-38%.
7. Name the indicator used in the determination of available chlorine in the sample of bleaching powder?
 Freshly prepared starch solution.
8. Give examples for chemical sanitizers.
 Sodium hypochlorite and bleaching powder are used as sanitizers.
9. Why freshly prepared starch is used as an indicator?
 To get the sharp end point, freshly prepared starch is used. If starch is not fresh, gives a reddish violet colour, which will be slowly decolorized by hypo solution.
10. What is the end point colour change?
 Blue to white precipitate.
11. Why blue colour appears, when starch is added?
 $\text{Starch} + \text{I}_2 \rightarrow (\text{C}_{24}\text{H}_{20}\text{O}_{20})\text{I}_3$ Blue colored complex.
12. What are the main constituents of bleaching powder?
 Calcium hypochlorite, Basic Calcium chloride and free calcium hydroxide.
13. Give two applications of bleaching powder.
 As disinfectant in potable water. To remove color stains.
14. Why starch indicator should be added towards the end point, not at the beginning?
 If I_2 is high at the beginning is strongly absorbed by the colloidal starch particles. It is not released even at the end point, though the solution becomes colorless.
15. Why iodometric titrations should not be carried out at high temperature?
 Iodine is volatile.
16. Why always excess of KI is used ?
 Because I_2 is very less soluble in water, but in presence of KI it forms an unstable complex salt KI_3 . This complex release iodine very slowly during titration due to reversible reaction.

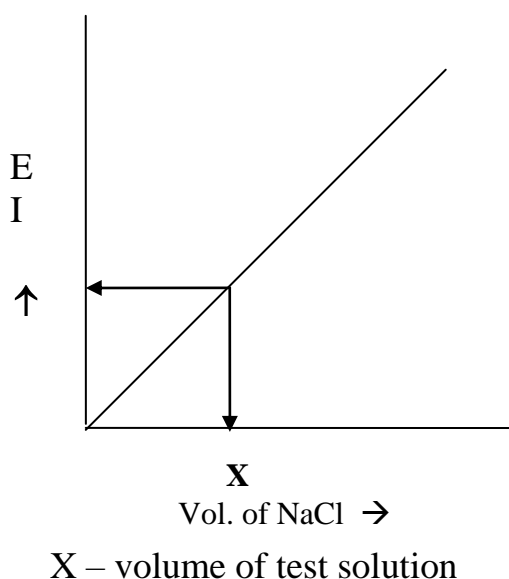


COLORIMETRIC ESTIMATION OF COPPER

Draw out 2, 4, 6, 8 & 10 ml of given copper sulphate solution into 5 separate 50 ml volumetric flasks. Add 5 ml ammonia to each of them & also into the test solution of unknown concentration. Dilute up to the mark using distilled water & mix well. Measure the absorbance of each of these standard solutions against blank solution (only ammonia & water) at 620 nm. Plot a graph of absorbance against volume of copper sulphate & determine the amount of copper in the test solution.

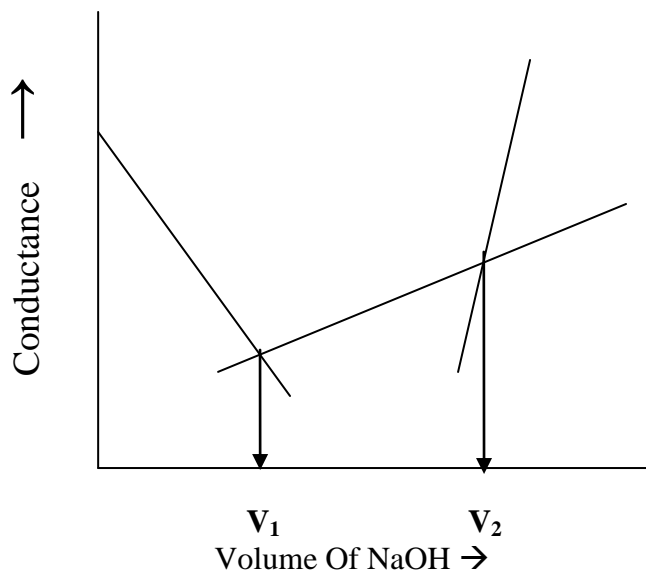
**FLAME PHOTOMETRIC ESTIMATION OF SODIUM IN THE GIVEN WATER SAMPLE**

Draw out 5,10,15,20 & 25 ml of given NaCl solution into 5 separate 50 ml volumetric flasks. Dilute up to the mark with distilled water & mix well. Measure the emission intensity at 589 nm. Plot a graph of emission intensity against volume of NaCl & determine the amount of Na in the test solution.



CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE USING STD. NaOH SOLUTION.

Pipette out 50 ml of acid mixture in to a beaker. Immerse the conductivity cell in to it & connect it to conductivity meter and measure the conductance. Add NaOH from the burette in the increments of 0.5 ml and measure the conductance after each addition. Plot a graph of conductance against volume of NaOH & determine the equivalence point. Calculate the normality & weight of HCl & CH₃COOH in the given solution.



Where, V_1 = Volume of NaOH required to neutralize HCl.
 $(V_2 - V_1)$ = Volume of NaOH required to neutralize acetic acid.

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

Pipette out 20 ml of given liquid into a wide limb of viscometer and suck the liquid through the other limb. Determine the time flow between 2 fixed points. One above & one below the bulb in the narrow limb of viscometer. Repeat and calculate the average time flow. Pour out the liquid, wash the viscometer with acetone and dry it. Now pipette out 20 ml of water into the wider limb and determine the average time flow for water as before. Determine the coefficient of viscosity of the given liquid using the formula.

$$\eta_L = \frac{\eta_w \times t_L \times d_L}{t_w \times d_w} \quad \text{millipoise.}$$

η_w = Viscosity coefficient of water

η_L = Viscosity coefficient of liquid

d_w = density of water

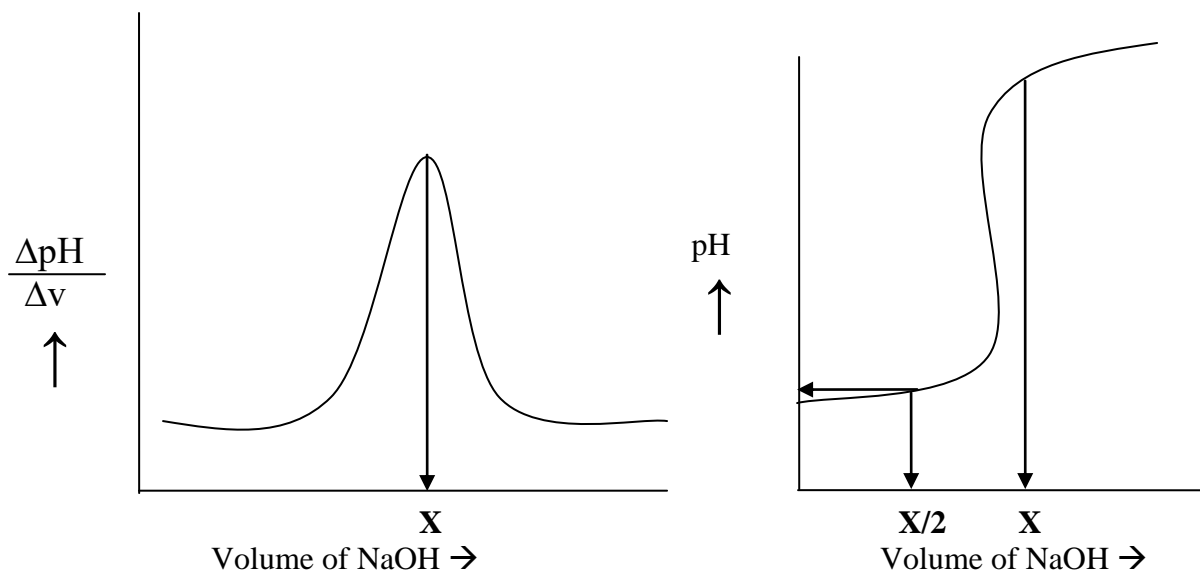
d_L = density of liquid

t_L = time flow of liquid and t_w = time flow of water.

DETERMINATION OF pK_a OF A WEAK ACID USING p^H METER

Pipette out 25 ml of the given weak acid into a beaker. Immerse the glass and calomel electrode assembly into it. Connect the electrodes to pH meter and measure the pH. Add NaOH from the burette in increments of 0.5 ml and measure the pH after each addition.

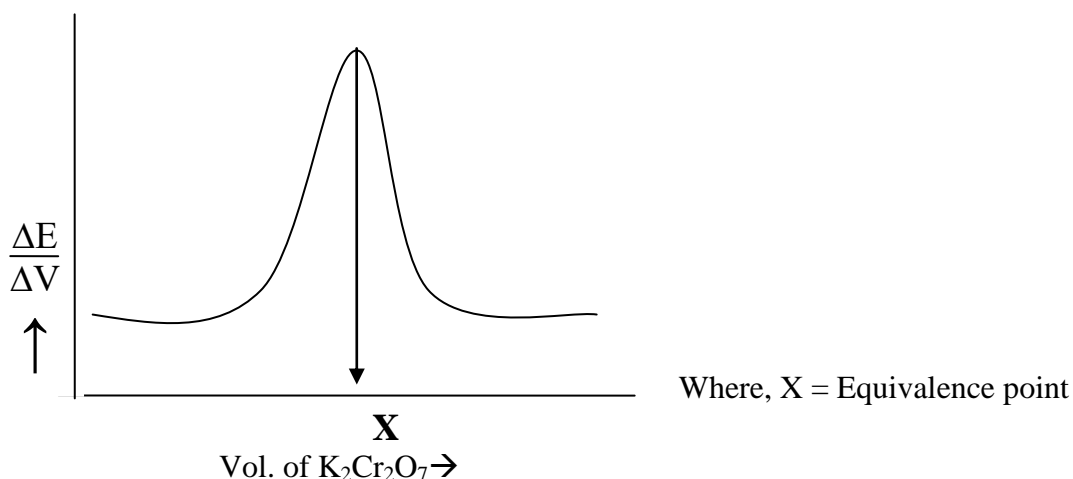
Plot a graph of $\Delta\text{pH} / \Delta V$ against volume of NaOH and determine equivalence point. Plot another graph of pH against volume of NaOH and determine pK_a value of the given weak acid.



Where X = Equivalence point; X/2 = Half Equivalence point

POTENTIOMETRIC TITRATION-ESTIMATION OF FAS USING STANDARD $K_2Cr_2O_7$ SOLUTION

Pipette out 25 ml of the given FAS solution into a beaker. Add one test tube of dilute sulphuric acid. Immerse the Platinum and calomel electrode assembly into it. Connect the electrodes to potentiometer and measure the potential. Add $K_2Cr_2O_7$ from the burette in increments of 0.5 ml and measure the potential after each addition. Plot a graph of $\frac{\Delta E}{\Delta V}$ against volume of $K_2Cr_2O_7$ and determine the equivalence point. From the normality of $K_2Cr_2O_7$, calculate the normality and weight of FAS present in the given solution.



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

Weigh out the given brass sample into a clean beaker. Add $\frac{1}{4}$ test tube of con. HNO_3 till brass dissolves completely. Add 1 test tube of distilled water and 1 gm of urea, boil till all brown fumes are expelled. Cool to room temperature. Transfer the solution in to a 250 ml volumetric flask and dilute up to the mark with distilled water and mix well.

- Burette : Standard Sodium thio sulphate solution
- Conical Flask : 25 ml Brass solution + NH_4OH drop wise till bluish
White ppt. + $\frac{1}{4}$ test tube dil CH_3COOH + 1 test tube of 10 % KI.
- Indicator : Starch - added towards the end point.
- Colour Change : Blue to white ppt.

From the volume of $Na_2S_2O_3$ Consumed, calculate the percentage of copper in the given brass sample.

ESTIMATION OF IRON IN HAEMATITE ORE SOLUTION USING STANDARD $K_2Cr_2O_7$ SOLUTION BY EXTERNAL INDICATOR METHOD.

Part A: Preparation of standard $K_2Cr_2O_7$ solution

Weigh out the given $K_2Cr_2O_7$ crystals accurately into a 250 ml volumetric flask. Dissolve in distilled water and dilute up to the mark, mix well.

$$\text{Normality of } K_2Cr_2O_7 = \frac{\text{Weight of } K_2Cr_2O_7 \text{ taken} \times 4}{\text{Equivalent weight of } K_2Cr_2O_7 (49)}$$

Part B: Estimation of Iron in haematite

Burette : Standard $K_2Cr_2O_7$ Solution

Conical Flask : 25 ml Haematite ore solution + 1/4th test tube of con. HCl, boil +
SnCl₂ drop wise till colorless. Cool & add 1/4th test tube HgCl₂

Indicator : Potassium ferricyanide

Color Change : Indicator fails to give any color with the drop of the test solution.

From the volume of $K_2Cr_2O_7$ consumed, calculate the percentage of iron in the given haematite ore solution.

ESTIMATION OF TOTAL HARDNESS OF WATER BY EDTA COMPLEXOMETRIC METHOD

Part A: Preparation of standard solution

Weigh out the given disodium salt of EDTA crystals accurately into a 250 ml volumetric flask. Add quarter test tube of ammonia. Dissolve in distilled water and dilute up to the mark, mix well.

$$\text{Molarity of EDTA} = \frac{\text{Weight of EDTA} \times 4}{\text{Molecular weight of EDTA (372)}}$$

Part B: Determination of total hardness

Burette : Standard EDTA Solution.

Conical Flask : 25 ml water sample + 3 ml Ammonia–ammonium chloride buffer solution.

Indicator : Eriochrome black-T.

Colour Change : Wine red to blue.

From the volume of EDTA consumed, calculate the total hardness of the given water sample.

ESTIMATION OF CaO IN CEMENT SOLUTION BY RAPID EDTA METHOD.**Part A: Preparation of standard solution**

Weigh out the given disodium salt of EDTA crystals accurately into a 250 ml volumetric flask. Add quarter test tube of ammonia. Dissolve in distilled water and dilute up to the mark, mix well.

$$\text{Molarity of EDTA} = \frac{\text{Weight of EDTA taken} \times 4}{\text{Molecular weight of EDTA (372)}}$$

Part B: Determination of % of CaO in cement solution

Burette : Standard EDTA Solution
 Conical Flask : 25 ml Cement solution + 5 ml Diethylamine + 5 ml Glycerol +
 10 ml NaOH
 Indicator : Patton & Reeder's
 Color Change : Wine red to blue

From the volume of EDTA consumed, calculate the percentage of CaO in the given Cement solution.

DETERMINATION OF COD OF WASTE WATER**Part A: Preparation of standard FAS solution (Mohr's salt solution)**

Weigh out the given FAS crystals accurately into a 250 ml volumetric flask. Add 2 test tube of H₂SO₄. Dissolve in distilled water and dilute up to the mark, mix well.

$$\text{Normality of FAS} = \frac{\text{Weight of FAS taken} \times 4}{\text{Equivalent weight of FAS (392)}}$$

Part B: Determination of COD of waste water sample.

Burette : Standard FAS Solution.
 Conical Flask : 25 ml waste water sample +10 ml K₂Cr₂O₇+ 1 test tube of 1:1 H₂SO₄
 Indicator : Ferroin
 Colour Change : Blush green to reddish brown.

Perform the blank titration in the same way as above, without taking waste water. From the difference of the titre values, calculate the COD of the given waste water sample.

DETERMINATION OF AVAILABLE CHLORINE IN BLEACHING POWDER**Model procedure:****Part –A :**

A stock solution of bleaching powder can be prepared by dissolving 1g of bleaching powder in 1 litre distilled water.

Part- B :

Burette: standard sodium thio sulphate solution.

Conical flask : 25ml bleaching powder solution + 5ml aceticacid + 10 ml KI.

Indicator : 1ml starch towards the end point.

End point: Blue to colourless

Perform the blank titration in the same way as above with out taking bleaching powder solution. From the titre value calucalate the % of availability of chlorine in the solution.

$$\% \text{ of availability of chlorine} = \frac{1000 \times B}{10}$$

Where B =

$$\frac{(V_1 - A_1) \text{ or } (V + A_4) \times N \times 35.46}{25}$$

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 Publisers.
3. Gary D. Christian, **“Analytical chemistry”**, 6th Edition, Wiley India.

