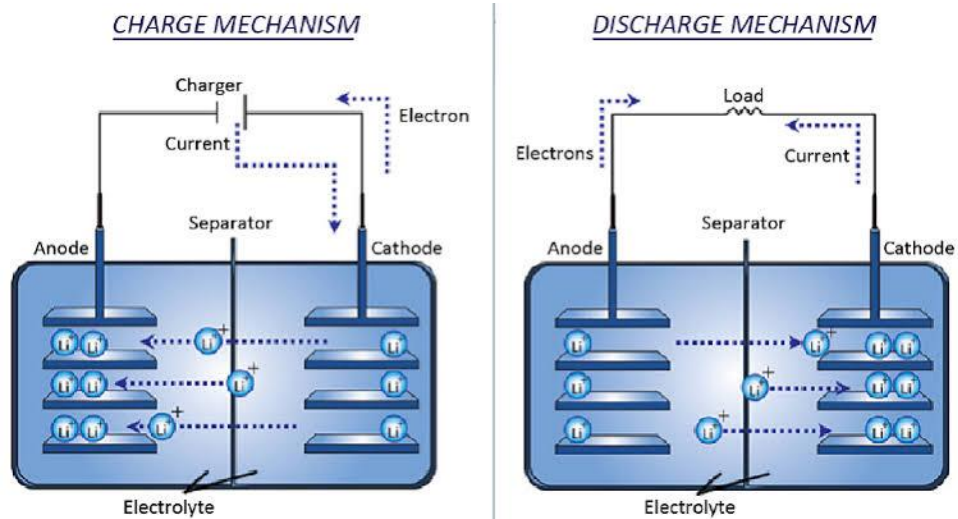


ENGINEERING CHEMISTRY

LABORATORY MANUAL



Li-ion battery

FOR STUDENTS OF I/II SEMESTER B.E

ATRIA INSTITUTE OF TECHNOLOGY

(As per new VTU Syllabus)

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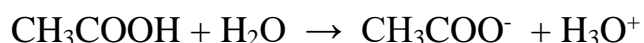
PART -A

1.DETERMINATION OF pK_a VALUE OF WEAK ACID USING PH METER

Aim: To determine the pK_a value of weak acid by pH titration

Principle:

Weak acids like acetic (CH₃COOH), formic acid (HCOOH) etc., undergoes partial dissociation and an equilibrium exists between ionized and unionized molecules of weak acid hence dissociation constants K_a can be defined for weak acid. Strong acids like hydrochloric acid (HCl) ionize completely in aqueous solution. The dissociation constant of weak (HA) like acetic acid can be written as.



$$K_a = \frac{[\text{CH}_3\text{COO}^-][\text{H}_3\text{O}^+]}{[\text{CH}_3\text{COOH}]}$$

Therefore pK_a = -log K_a

Higher the value of pK_a, lower is the K_a value of acid and therefore weaker is the acid. pK_a value of weak acid can be determined by the change in pH.

In the titration of acid with base, the pH of the solution gradually increases, and equivalence point is indicated by sharp increases in pH (Fig.1). The equivalence point is determined by plotting a graph of volume of base against ΔpH/ΔV (Fig.2)

During the titration of weak acid with base, salt of the weak acid is formed. For such an equilibrium, according to Henderson-Hasselbalch equation.

$$\text{pH} = \text{pK}_a + \log \frac{[\text{salt}]}{[\text{acid}]}$$

At half equivalence point 50% of acid is converted into its salt and therefore

$$[\text{acid}] = [\text{salt}]$$

then, pH = pK_a

therefore pH, at half equivalence point gives the pK_a, value of weak acid.

Procedure

Pipette out 25cm^3 of the given acid into a beaker and add one test tube of distilled water. Immerse the glass electrode and calomel electrode assembly into it. Connect the electrode to pH meter. Fill the burette with sodium hydroxide. Now add sodium hydroxide from the burette in increments of 0.5cm^3 and measure pH. Continue the addition of sodium hydroxide till there is slight increases in pH on the addition base. Plot a graph of $\Delta\text{pH}/\Delta V$ against volume of sodium hydroxide added (graph2). Determine the equivalence point. Plot another graph of pH against volume of sodium hydroxide and determine the pK_a value of weak acid from the graph as shown in graph 1 and graph 2.

Result:

The pK_a value of the given weak acid from the graph =.....

OBSERVATION AND CALCULATION

Burette: Sodium hydroxide solution

Electrodes: Glass electrode and Calomel electrode

Beaker: 25 cm³ of weak acid+ 1 test tube of distilled water.

Volume of NaOH cm ³	pH	ΔV	ΔpH	$\Delta pH/\Delta V$
0.0		NA	NA	NA
0.5		0.5		
1.0		0.5		
1.5		0.5		
2.0				
2.5				
3.0				
3.5				

[LHS NEED TO BE WRITE IN RECOED]

According to Henderson-Hasselbalch equation.

$$\text{pH} = \text{pK}_a + \log \frac{[\text{salt}]}{[\text{acid}]}$$

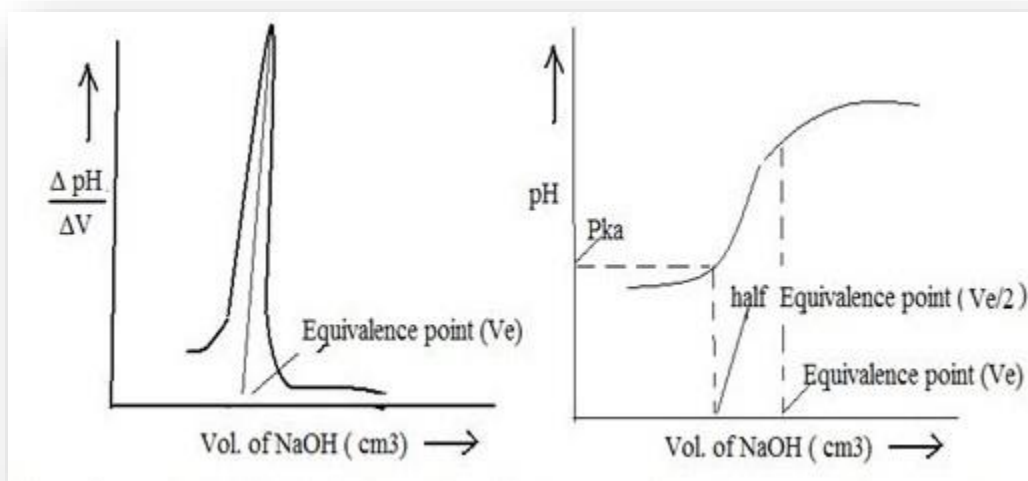
At half equivalence point 50% of acid is converted into its salt and therefore

$$[\text{acid}] = [\text{salt}]$$

then, $\text{pH} = \text{pK}_a$

Graph (1)

Graph (2)



From the graph (2) full equivalence point is $V_{\text{equivalence point}} = \dots\dots\dots \text{ml}$

Half equivalence point is $= V_{\text{eq}}/2 = \dots\dots\dots \text{ml}$

Therefore, from graph (1) at half equivalence, $\text{pH} = \text{pK}_a = \dots\dots\dots$

[LHS NEED TO BE WRITE IN RECOED]

2. ESTIMATION OF FAS USING STANDARD $K_2Cr_2O_7$ BY POTENTIOMETRIC METHOD.

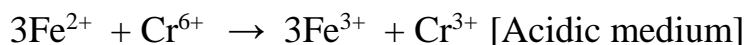
Aim: To estimate the amount of ferrous ammonium sulphate using standard potassium dichromate potentiometrically.

Principle:

A potentiometric titration involves the measurement of change in potential with a suitable electrode which responds to the change in concentration during the titration. The electrode which responds to the change in concentration of ion in the solution is called indicator electrode. The indicator electrode is combined with reference electrode (whose potential does not change during the titration) to form a cell and e.m.f of the cell changes gradually till the end point and changes rapidly at very close to the end point and again the change is gradual after the end point.

A plot the first derivative curve, in which $(\Delta E/\Delta V)$ is plotted against V, the peak of the curve gives the end point of the titration.

The potentiometric titration of FAS (Mohr's salt) solution with $K_2Cr_2O_7$ in the presence of H_2SO_4 is a redox titration.



The presence of oxidized and reduced form of the same substance in a solution gives rise to the formation of an oxidation-reduction electrode, developing an electrode potential, which can be picked up by dipping a Platinum wire. Thus, when the titration is commenced, both Fe^{2+} and Fe^{3+} ions are present in the solution, developing an electrode potential which is picked up by a Pt wire. Thus $(Pt/Fe^{3+}, Fe^{2+})$ is the indicator electrode, whose electrode potential is given by,

$$E = E^0 + \frac{2.303 RT}{nF} \log \frac{(Fe^{3+})}{(Fe^{2+})}$$

During the titration (Fe^{3+}) goes on increasing and (Fe^{2+}) goes on decreasing as $K_2Cr_2O_7$ solution is added continuously, gradually changing the potential. Near the end point the ration (Fe^{3+}/ Fe^{2+}) increases rapidly as (Fe^{2+}) becomes very small and tending to zero at the end point. Thus, the e.m.f increases rapidly at very close to the end point. When Fe^{2+} is completely converted into Fe^{3+} by $K_2Cr_2O_7$, the electrode, $(Pt/Fe^{3+}, Fe^{2+})$ ceases to exist. But the presence of slight excess of $K_2Cr_2O_7$, brings in

existence of (Pt/Cr⁶⁺, Cr³⁺) electrode. Thus, after the end point, it is the electrode potential of the (Pt/Cr⁶⁺, Cr³⁺) electrode, which is going to change. Because of both the factors i.e., increase in the value of (Fe³⁺/ Fe²⁺) and change of electrode from (Pt/Fe³⁺, Fe²⁺) to (Pt/Cr⁶⁺, Cr³⁺) there is a large change in potential at the end point.

Procedure:

Pipette out 25 cm³ of ferrous ammonium sulphate into a beaker. Add two test tubes of dilute sulphuric acid. Immerse calomel and platinum electrode assembly into it. Connect the platinum and calomel electrode to a potentiometer and measure the potential. Fill the burette with 0.5 N potassium dichromate solution. Add 0.5 cm³ of potassium dichromate to the beaker. Stir the solution and note down the potential. Continue the procedure till the potential shows the tendency to increase rapidly. Now add potassium dichromate in increments of 0.5 cm³ and measure the potential after each addition. Plot a graph of $\Delta E/\Delta V$ against volume of potassium dichromate and determine the equivalence point from the graph. Calculate the normality and weight of ferrous ammonium sulphate in the given solution.

Result:

1. Normality of the given FAS solution = $N_{FAS} = \dots\dots\dots$ N
2. Weight of FAS in one liter of solution = $\dots\dots\dots$ g/liter

OBSERVATION AND CALCULATIONS

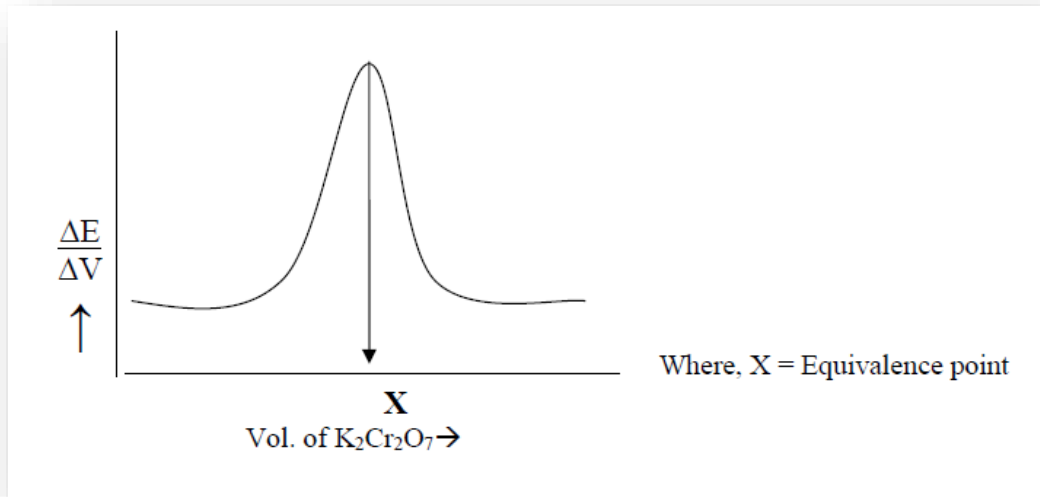
Burette: 0.5 N $\text{K}_2\text{Cr}_2\text{O}_7$ solution

Electrodes: Platinum electrode and calomel electrode

Beaker: 25 cm^3 of FAS solution + 2 test tubes of dilute H_2SO_4

Volume of $\text{K}_2\text{Cr}_2\text{O}_7$ (cm^3)	E (mv)	ΔV	ΔE	$\Delta E/\Delta V$
0.0		-----	-----	-----
0.5				
1.0				
1.5				
2.0				
2.5				

[LHS NEED TO BE WRITE IN RECOED]



From the graph, the volume of $K_2Cr_2O_7$ solution = $V_{K_2Cr_2O_7} = \dots\dots\dots$

$$N_1 V_1 = N_2 V_2$$

$$(N \times V)_{FAS} = (N \times V)_{\text{potassium dichromate}}$$

$$N_{FAS} = \frac{(N \times V)_{\text{potassium dichromate}}}{V_{FAS}}$$

$$N_{FAS} = \frac{0.5 \times \text{from graph}}{25}$$

$$N_{FAS} = \dots\dots\dots N$$

$$\begin{aligned} \text{Therefore, weight of FAS in a liter of solution} &= N_{FAS} \times \text{Eq. Wt. of FAS (392)} \\ &= N_{FAS} \times (392) \\ &= \dots\dots\dots \text{ g/ltr.} \end{aligned}$$

[LHS NEED TO BE WRITE IN RECOED]

3. CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE USING SODIUM HYDROXIDE

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

Principle:

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 steep increase in conductance due to the ions furnished by strong base.

Procedure:

Pipette out 25 cm^3 of acid mixture in a 250 ml 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 std. NaOH from the burette in the increments of 0.5 cm^3 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 neutralize HCl. By knowing the normality & volume of NaOH calculate the normality & amount of HCl & CH_3COOH .

Result:

- (i) Normality of HCl = $N_{HCL} = \dots\dots\dots N$
- (ii) Normality of $CH_3COOH = N_{CH_3COOH} \dots\dots\dots N$
- (iii) Amount of HCl per liter of solution..... g/liter
- (iv) Amount of CH_3COOH per liter of solution..... g/liter

OBSERVATION AND CALCULATION

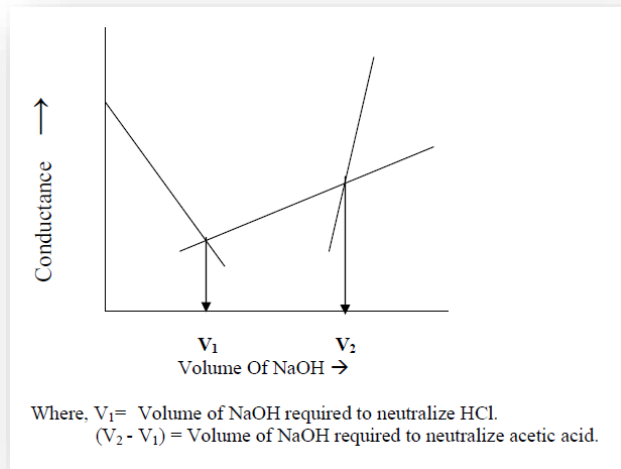
Burette: 0.5 N NaOH solution

Electrode: Conductivity cell (Platinum electrode)

Beaker: 25cm³ of acid mixture + 1 test tube distilled water

Volume of NaOH (cm ³)	Conductance Ohm ⁻¹ cm ⁻¹
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	

[LHS NEED TO BE WRITE IN RECOED]



V1 = Neutralization point of HCl

V2 = Neutralization point of CH₃COOH

$$(NV)_{\text{HCl}} = (NV)_{\text{NaOH}}$$

$$N_{\text{HCl}} = \frac{(N \times V)_{\text{NaOH}}}{V_{\text{HCl}}}$$

$$= \frac{0.5 \times \text{from graph } (V_1)}{25} = \dots\dots\dots N$$

$$\begin{aligned} \text{Weight of HCl in one liter of the solution} &= N_{\text{HCl}} \times \text{Eq.wt. of HCl (36.5)} \\ &= \dots\dots\dots \text{g/dm}^3 \end{aligned}$$

$$\begin{aligned} N_{\text{CH}_3\text{COOH}} &= \frac{(N \times V)_{\text{NaOH}}}{V_{\text{acetic acid}}} \\ &= \frac{0.5 \times \text{from graph } (V_2 - V_1)}{25} \\ &= \dots\dots\dots N \end{aligned}$$

$$\begin{aligned} \text{Weight of CH}_3\text{COOH/ liter of solution} &= N_{\text{CH}_3\text{COOH}} \times \text{Eq.wt. of CH}_3\text{COOH (60)} \\ &= \dots\dots\dots \text{g/dm}^3 \end{aligned}$$

[LHS NEED TO BE WRITE IN RECOED]

4. COLORIMETER ESTIMATION OF COPPER

Aim: To estimate the amount of copper present in the given solution by colorimetric method

Principle:

Colorimetric analysis is based on measurement of quantity of light absorbed by a colored solution. Beer-Lambert's law forms the basis of this experiment. When a monochromatic light of intensity I_o is incident on a transparent medium, a part I_a is absorbed, a part I_r is reflected and remaining part. It is transmitted.

Therefore, $I_o = I_a + I_r + I_t$

For glass-air I_r is negligible, therefore $I_o = I_a + I_t$

The ratio $I_t/I_o = T$ is called transmittance, $\log (1/T) = \log (I_o/I_t) = A$, called absorbance or optical density.

The relation between absorbance A , concentration c (in moles per liter) and path length l (in cm) is given by Beer's-Lambert's law

$$A = \text{Log} (I_o/I_t) = \epsilon cl$$

Where I_o = intensity of the incident light, I_t = Intensity of the transmitted light

c = concentration of colored solution, l = thickness of the colored medium,

ϵ = molar absorption coefficient

So, the same result can be obtained by plotting $\log T$ against concentration also.

Cupric ions (Cu^{2+}) react with ammonia to give a deep blue a deep blue colour due to the formation of cuprammonium complex ion.



A Series of standard solution containing cupric ions are treated with ammonia and the absorbance of each of these solutions is measured at 620 nm (λ_{max}) at which solution shows maximum absorbance. A calibration curve is obtained by plotting concentration against absorbance or $\log T$. The given unknown solution is also treated with ammonia in the same way as the standard and its absorbance is measured. Its concentration is determined from the calibration curve.

Procedure:

Pipette out 2,4,6,8 and 10 cm³ of the given copper sulphate solution (containing 8g of copper per litre) into standard volumetric flasks add 5 cm³ of ammonia solution into each of them and make up to the mark with distilled water and shake well.

Select a filter (620nm) in the colorimeter and adjust the initial readings to zero absorbance with blank solution (distilled water +ammonia) using a glass cuvette. Fill the glass cuvette with the standard of lowest concentration and measure the absorbance. Repeat for other standard solutions also. Plot absorbance (Y-axis) against volume (X-axis) of copper sulphate solution taken.

For the given unknown add 5 cm³ of ammonia and make up to the mark with distilled water. Measure the absorbance using the colorimeter at the same wavelength as said above. From the graph, determine the concentration of the unknown solution.

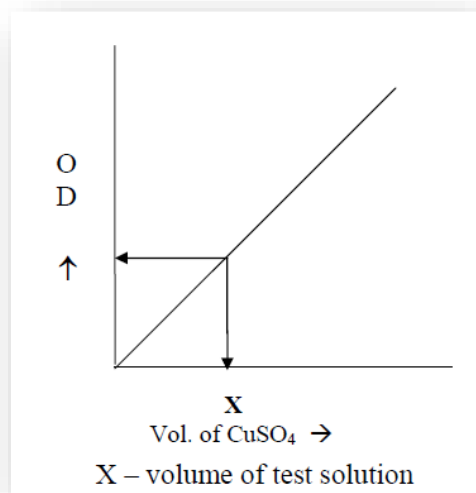
Result:

- 1.The volume of unknown solution from the graph=..... ml
- 2.Weight of copper (II) present in the given solution=..... mg/ltr.

OBSERVATION AND CALCULATION

Wavelength of monochromatic light used for blue colour = $\lambda_{\max} = 620\text{nm}$

Flask No.	Volume of copper sulphate (cm^3)	Absorbance (Optical density)
1.	$2\text{ cm}^3 + 5\text{ ml.NH}_3 + \text{H}_2\text{O}$	
2	$4\text{ cm}^3 + 5\text{ ml.NH}_3 + \text{H}_2\text{O}$	
3	$6\text{ cm}^3 + 5\text{ ml.NH}_3 + \text{H}_2\text{O}$	
4	$8\text{ cm}^3 + 5\text{ ml.NH}_3 + \text{H}_2\text{O}$	
5	$10\text{ cm}^3 + \text{c}$	
6	Test Solution (unknown + $5\text{ ml.NH}_3 + \text{H}_2\text{O}$)	
7	Blank ($5\text{ ml.NH}_3 + \text{H}_2\text{O}$)	



[LHS NEED TO BE WRITE IN RECOED]

249.54 g of CuSO_4 contains 63.54 g of Cu

Therefore given 8 g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ solution contains $\frac{63.54 \times 8}{249.54} = 2.037$ g of Cu

1000 cm^3 of stock solution 2.037 g of copper

Therefore, the graph (V_{Unknown}) cm^3 solution = $\frac{(V_{\text{test. solution}}) \times 2.037}{1000}$
=g of Cu
= $\times 10^3$ mg of Cu

[LHS NEED TO BE WRITE IN RECOED]

5. DETERMINATION OF VISCOSITY CO-EFFICIENT OF A GIVEN LIQUID USING OSTWALD'S VISCOMETER.

Aim: To determine viscosity co-efficient of a given organic liquid using Ostwald's Viscometer.

Principle:

Viscosity is a property of liquid, defined as the resistance offered towards the flow of a layer of liquid over the other successive layer. Viscosity arises due to internal friction between moving layers of molecules. A liquid flowing through a cylinder tube of uniform diameter is expected to move in the form of molecular layers. A layer close to the surface is almost stationary while layer at the axis of tube moves faster than any other intermediate layer. The co-efficient of viscosity (η) is defined as tangential force per unit area required to maintain unit velocity gradient between any two successive layers of a liquid situated unit distance apart. The viscosity co-efficient of liquid is given by Poiseuille's formula.

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

Where P= Pressure difference between the two ends of the tube= $h d g$,

h = height

d= density

g= acceleration due to gravity

r= radius of the capillary,

v= volume of the liquid,

l= length of the capillary tube,

t= time required for the flow.

If equal volumes of two different liquids are allowed to flow through the same capillary tube under identical condition. Then relative viscosity is given by the expression,

$$\eta_1 = (t \times d)_{\text{liquid}}$$

$$\eta_2 = (t \times d)_{\text{water}}$$

The time (t_1) taken by the given liquid to flow through a certain distance in capillary is determined. The time (t_w) taken by water to flow through the same distance is measured. Viscosity co-efficient of a given liquid is calculated from the densities

(d_l and d_w) of the liquid and water and viscosity co-efficient of water (η_{water}). The viscosity measurement is carried out in Ostwald's viscometer.

Since the viscosity is dependent inversely on temperature, the experiment is carried out constant temperature. The viscometer should be immersed vertically in a beaker containing water, which maintains constant temperature (called thermostat), throughout the determination.

Procedure:

Take a clean and dry Ostwald's viscometer. Fix it vertically to a stand in a constant water bath (beaker containing water). Transfer 20 cm³ of the given liquid into the wider limb Ostwald's viscometer using a pipette. Suck the given liquid above the upper mark of the viscometer allow the liquid to flow freely through the capillary. When the level of the liquid just crosses the upper mark start the stop clock and stop the stop clock when the level of the liquid just crosses the lower mark of the viscometer. Note the time of flow in seconds (t_{liquid}). Repeat the same procedure to get agreeing values.

Pour out the liquid rinse the viscometer with acetone and dry it. Transfer 20 cm³ of water in to the wider limb of the Ostwald's viscometer, as described above note the time (t_{water}) taken by water to flow through the same distance in the viscometer thoroughly before conducting the experiment with the test liquid and distilled water.

Result:

Viscosity co-efficient of the given liquid=..... Millipoise at.....⁰C

OBSERVATION AND CALCULATIONS

Constant temperature water bath (thermostat) temperature =⁰C

To determine the flow time

	Time of flow (seconds)			Average time of flow
	Trial 1	Trail 2	Trial 3	
Given organic Liquid (t _l)				
Water (t _w)				

$$\eta_{\text{liquid}} = \dots\dots\dots \text{ Millipoise}$$

Where density of liquid = $d_{\text{liquid}} = \dots\dots\dots \text{ g/cm}^3$

Where density of water = $d_{\text{water}} = \dots\dots\dots \text{ g/cm}^3$

Viscosity co-efficient of water = $\eta_{\text{water}} = \dots\dots\dots \text{ millipoise}$

Therefore, co-efficient of the liquid $\eta_{\text{liquid}} = \frac{(t \times d)_{\text{liquid}}}{(t \times d)_{\text{water}}} \times \eta_{\text{water}}$

$$= \dots\dots\dots \text{ Millipoise}$$

[LHS NEED TO BE WRITE IN RECOED]

PART -B

1.DETERMINATION OF TOTAL HARDNESS OF WATER USING EDTA

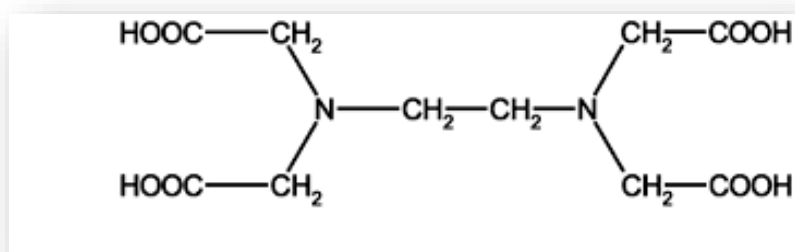
Aim: To determine the total hardness of the given sample of water using disodium salt of EDTA

Principle:

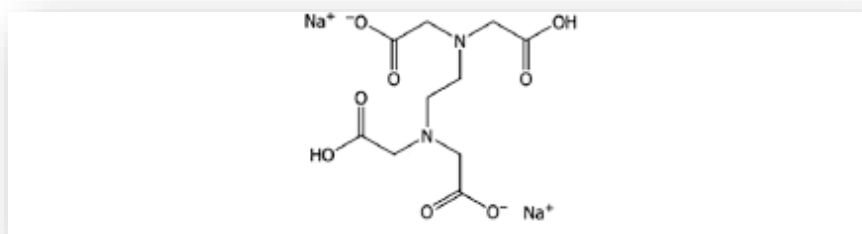
Hard water does not give lather easily with soap, as the hardness causing ions react with soap to form insoluble soap. Hardness of water is due to presence of dissolved salts of calcium, magnesium and other metal ions. Temporary hardness is due to bicarbonates of calcium and magnesium, which can be removed by boiling the water. On boiling, bicarbonates are converted into insoluble carbonates of calcium and magnesium. Permanent hardness is due to the presence of chlorides and sulphates of calcium, magnesium and other metals, which is removed only by chemical treatment. The sum of temporary hardness and permanent hardness is known as total hardness. The hardness is expressed in parts per million (ppm) equivalent of calcium carbonate or mg/litre.

Hardness of water is determined by titrating a known volume of hard water sample with Na_2EDTA , using Eriochromo black-T (EBT) as indicator, in presence of ammonium hydroxide-ammonium chloride buffer (pH=10). Ethylene diamine tetra acetic acid (EDTA) or its disodium salt (Na_2EDTA) is a strong complexing agent has the following structure.

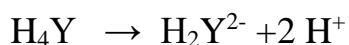
Structure of EDTA



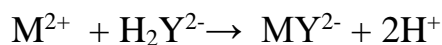
Structure of Na₂EDTA



The ionization of EDTA in solution is represented as



The anion formed in the above ionization forms complexes with metal ions M^{2+} which can be given as,



When few drops of the indicator EBT is added, metal ions (Ca^{2+} , Mg^{2+}) of hard water forms a wine red colored complex with the indicator.



When the solution is titrated Na₂EDTA reacts, preferentially with free metal ions (Ca^{2+} , Mg^{2+}) present in the solution. Near the end point, when the free metal ions are exhausted in the solution, further addition of Na₂EDTA dissociates the M-EBT complex, consumes the metal ions and release free indicator, which is blue in color. Therefore, the color change is wine red to clear blue.

The indicator, EBT shows the colour change at pH range 9-11 therefore the pH of the solution is maintained around 10 using ($\text{NH}_4\text{OH} + \text{NH}_4\text{Cl}$) buffer. EDTA being sparingly soluble in water, generally its disodium salts (Na₂EDTA) is used which readily dissolves in water. A standard solution of Na₂EDTA is prepared by dissolving a known weight of Na₂EDTA in distilled water and making up to 250ml.

Molecular weight of disodium salt of Na₂EDTA = 372.24

Therefore, Molarity of Na₂EDTA solution = $\frac{\text{Wt. of Na}_2\text{EDTA taken} \times 4}{\text{Mol.Wt. Na}_2\text{EDTA (372.24)}}$

Procedure:

(a) Preparation of standard solution of Na₂EDTA:

Weigh accurately given Na₂EDTA crystals and transfer it into a 250 cm³ standard volumetric flask. Add 5 cm³ of ammonia and add ion-exchange ware to dissolve the crystals. Make up the solution to 250cm³ and mix well for uniform concentration.

(b) Determination of total hardness of water:

Pipette out 25cm³ of given hard water sample into a clean conical flask. Add 5cm³ of buffer (NH₄OH + NH₄Cl) of pH=10 and 2-4 drops of EBT indicator. Titrate this solution against standard Na₂EDTA solution taken in a burette, until the colour of the solution changes from wine to clear blue. Repeat the titration for concordant values.

Result: Total Hardness of the given sample of hard water =..... ppm
(parts per million) of CaCO₃.

OBSERVATION AND CALCULATIONS

I Preparation of standard Na₂EDTA solution:

Weight of weighing bottle + Na₂EDTA = W₁ = g

Weight of empty weighing bottle = W₂ = g

Therefore, weight of Na₂EDTA = (W₁ - W₂) = W₃ g

$$\begin{aligned} \text{Therefore, molarity of Na}_2\text{EDTA} = M_{\text{Na}_2\text{EDTA}} &= \frac{\text{Wt. of Na}_2\text{EDTA taken} \times 4}{\text{Mol. Wt. of Na}_2\text{EDTA (372.24)}} \\ &= \frac{W_3 \times 4}{372.24} \\ &= \dots\dots\dots \text{M} \end{aligned}$$

II Determination of total hardness of water:

Burette: Std. Na₂EDTA solution.

Conical flask: 25cm³ hard water sample + 5 cm³ buffer solution
(NH₄OH + NH₄Cl) of pH=10

Indicator: Eriochromo black -T

End point: Wine Red to clear Blue

Burette level	I	II	III
Final burette reading			
Initial burette reading			
Volume of Na ₂ EDTA (cm ³) Consumed.			

Volume of Na₂EDTA consumed = V_{Na₂EDTA} =cm³

[LHS NEED TO BE WRITE IN RECOED]

Since $M_1 V_1 = M_2 V_2$

$$(M \times V)_{\text{Na}_2\text{EDTA}} = (M \times V)_{\text{Hard water}}$$

Therefore, $M_{\text{hard water}} = \frac{(M \times V)_{\text{Na}_2\text{EDTA}}}{V_{\text{Hard water}}}$

$$\begin{aligned} &= \frac{(M \times V)_{\text{Na}_2\text{EDTA}}}{25} \\ &= \dots\dots\dots M \end{aligned}$$

Therefore, Wt. of CaCO_3 / litre of solution = $W_4 = M_{\text{hard water}} \times \text{Mol. Wt. of CaCO}_3$
(100)

$$= \dots\dots\dots \text{g/dm}^3$$

Therefore, $W_4 = \dots\dots\dots \text{g/dm}^3$

Total hardness of water in ppm = $\frac{W_4 \times 10^6}{10^3}$

$$\begin{aligned} &= \dots\dots\dots \text{ppm of CaCO}_3 \end{aligned}$$

[LHS NEED TO BE WRITE IN RECOED]

2. DETERMINATION OF CALCIUM OXIDE IN CEMENT USING EDTA

Aim: To determine the percentage of calcium oxide (CaO) in the given sample of cement solution using Na₂EDTA

Principal:

Composition of cement: 50-60% CaO, 21% SiO₂, MgO, Al₂O₃ and Fe₂O₃.

Cement contains oxides of calcium, aluminum, magnesium, iron is filtered. On treating with ammonia, aluminum and iron can be precipitated as their hydroxides and can be removed by filtration. Therefore, the calcium contents in the given solution, a known volume of its titrated with standard Na₂EDTA by using Patton & Reeder's indicator at pH of 12 to 14. The indicator combines with calcium ions forming a wine-red colored complex.



(Wine red)

When the solution is titrated, Na₂EDTA react with Ca²⁺ ions present in the cement solution. Near the end point, when free calcium ions are exhausted in the solution, further addition of Na₂EDTA dissociates Ca-IN complex, consumes the calcium ions and leaves free indicator, which is blue in color. Therefore, the colour change is wine red to clear blue.

The interference of magnesium ions is prevented by precipitating magnesium ions is prevented by precipitating magnesium as magnesium hydroxide. This is done by adding 4N NaOH and pH of the solution is maintained to pH at 12.5 by adding 5 cm³ of Diethylamine. Glycerol is added to get the sharp endpoint. The standard Na₂EDTA solution is prepared by dissolving a known weight of Na₂EDTA in a known volume of the solution, using distilled water.

Molecular weight of disodium salt of Na₂EDTA = 372.24

Procedure:

(a) Preparation of standard of Na₂EDTA:

Weigh accurately given of Na₂EDTA crystals and transfer it into a 250cm³ standard volumetric flask. Add 5cm³ of ammonia and add ion-exchange water to dissolve the crystals. Make up the solution to 250cm³ and mix well for uniform concentration.

(b) Determination of calcium oxide in cement solution:

Pipette out 25cm³ of given cement solution into a clean conical flask using a pipette. Add 5 cm³ of 4N Sodium hydroxide to it. Add 5 cm³ of Diethylamine and glycerol to the resulting solution. Allow the solution to stand for 3-5 minutes with occasional stirring. Add 3-4 drops of Patton Reeder's indicators and titrate against Na₂EDTA solution taken in the burette till the colour changes from wine red to clear blue. Repeat for agreeing values.

Result:

The percentage of CaO in the given cement solution=.....

OBSERVATION AND CALCULATIONS

I Preparation of standard Na₂EDTA solution:

Weight of weighing bottle + Na₂EDTA = W₁ = g

Weight of empty weighing bottle = W₂ = g

Therefore, weight of Na₂EDTA = (W₁-W₂) = W₃.....g

$$\begin{aligned} \text{Therefore, molarity of Na}_2\text{EDTA} = M_{\text{Na}_2\text{EDTA}} &= \frac{\text{Wt. of Na}_2\text{EDTA taken} \times 4}{\text{Mol.Wt. of Na}_2\text{EDTA (372.24)}} \\ &= \frac{W_3 \times 4}{372.24} \\ &= \dots\dots\dots \text{M} \end{aligned}$$

II Determination of CaO:

Burette: Std. Na₂EDTA solution.

Conical flask: 25cm³ cement solution + 3/4th test tube 4N NaOH+ 5 cm³ glycerol + 5 cm³ Diethylamine

Indicator: Patton Reeder's

End point: Wine Red to clear Blue

Burette level	I	II	III
Final burette reading			
Initial burette reading			
Volume of Na ₂ EDTA (cm ³) Consumed.			

Volume of Na₂EDTA consumed = V_{Na₂EDTA} = cm³

[LHS NEED TO BE WRITE IN RECOED]

Since $M_1V_1=M_2V_2$

$$(M \times V)_{\text{Na}_2\text{EDTA}} = (M \times V)_{\text{cement solution}}$$

$$\begin{aligned} \text{Therefore, } M_{\text{hard water}} &= \frac{(M \times V)_{\text{Na}_2\text{EDTA}}}{V_{\text{cement solution}}} \\ &= \frac{(M \times V)_{\text{Na}_2\text{EDTA}}}{25} \end{aligned}$$

$$M_{\text{cement solution}} = \dots\dots\dots M$$

Therefore Wt. of CaO/ litre of cement solution = $W_4 = M_{\text{cement solution}} \times \text{Mol. Wt. of CaO (56.08)}$

$$= \dots\dots\dots \text{g/dm}^3$$

Therefore $W_4 = \dots\dots\dots \text{g/dm}^3$

Given (X) $\dots\dots\dots$ g cement contains $W_4 = \dots\dots\dots$ g of CaO

Therefore 100 g of cement contains = $\frac{W_4 \times 100}{X}$

$$\begin{aligned} &X \\ &= \dots\dots\dots \text{ g of CaO} \end{aligned}$$

[LHS NEED TO BE WRITE IN RECOED]

3. DETERMINATION OF COPPER IN BRASS

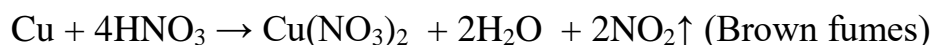
Aim: To determine the percentage of copper in the given sample of brass by iodometric titration

Principle:

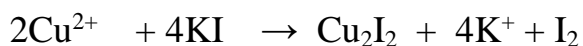
Brass is an alloy of copper and Zinc. It also contains small amounts of Tin, Lead and Iron. The percentage composition of a typical brass alloy is

Cu: 50-90% Zn: 20-40% Sn: 0-6% Pb:0-2% Fe: 0-1%

The amount of copper present in brass can be determined iodometrically. A solution of brass is made by dissolving the sample in minimum amount of nitric acid. Copper present gets converted to its nitrate with the evolution of oxides of nitrogen.

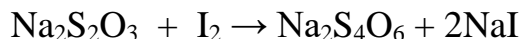


Adding urea destroys oxides of nitrogen present in the brass solution. The mineral acid (HNO_3) present in the brass solution is neutralized by adding ammonium hydroxide (NH_4OH) till a pale blue precipitate of cupric hydroxide $[\text{Cu}(\text{OH})_2]$ is obtained. Otherwise, being an oxidizing agent, nitric acid may also liberate iodine from added potassium iodide during titration. The $\text{Cu}(\text{OH})_2$ precipitate, is dissolved with dilute acetic acid. Other mineral acids are not preferable, as they bring down the pH to a very small value, at which liberation of Iodine from potassium iodide by copper ion is not quantitatively. When potassium iodide is added, it liberates iodine due to the reduction of cupric ions and yellow precipitate of cuprous iodide (Cu_2I_2) is formed.



Cuprous iodide

The liberated iodine is titrated against sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) using starch as indicator. Starch forms a blue colored complex with iodine. At the end point when free iodine is exhausted, added quantity of $\text{Na}_2\text{S}_2\text{O}_3$ dissociates starch - iodine complex, consumes iodine and liberates starch, thereby discharging the blue colour. A white precipitate of sodium tetrathionate is formed. Therefore, the end point is discharge of blue colour to white colour.



Sodium tetrathionate

Starch indicator is added near the end point and not in the beginning for the reasons that (i) at high concentration of iodine (I_2), starch may form an insoluble starch- I_2 complex and (ii) the I_2 also gets absorbed on colloidal starch particles in starch solution. The absorbed iodine cannot be liberated by the addition of sodium thiosulphate, and the amount of iodine getting absorbed is more when iodine concentration is high. Thus, adding starch near the end point minimizes the error.

During the liberation of I_2 from KI, Cu^{2+} gets reduced to Cu^{+} with a change in oxidation state by +1.

Thus, equivalent weight of Cu = atomic weight = 63.54

Procedure:

Weight accurately given brass sample and transfer it into a clean conical flask. Dissolve the alloy in 10-12 drops of concentrated nitric acid. Then add about a gram of urea and 2 test tube of distilled water, boil for 2-3 minutes. Cool the mixture. Add NH_4OH solution dropwise until a bluish white precipitate persists. Add dilute acetic acid dropwise just to dissolve the precipitate and then add about 3-5 drops in excess followed by the addition of 10 cm^3 of 10% KI solution. Titrate the liberated iodine against sodium thiosulphate solution taken in the burette until the color of the solution changes to pale yellow. At this stage add starch solution (at a stretch) as indicator and continue the titration till the blue colour changes to white Repeat the titration for concordant values.

Result:

1. Percentage of copper in the given sample brass sample (1) =

OBSERVATION AND CALCULATIONS

Preparation of Brass Solution:

Weigh the given brass sample. Add 10-12 drops of con. HNO_3 dissolve Add 1 gm of urea and 2 test tube of water, cool.

1. Weight of brass sample (1) = $W_1 = \dots\dots\dots$ g

Burette: Std. $\text{Na}_2\text{S}_2\text{O}_3$ solution (0.05N)

Conical flask: Above brass solution+ add NH_4OH dropwise to get bluish white ppt.+ Acetic acid to get clear solution+ 3 drops excess + add 1 test tube of 10% KI solution

Indicator: Starch (at a stretch) near the end point.

End point: Disappearance of Blue colour to White colour.

Burette level	I	II	III
Final burette reading			
Initial burette reading			
Volume of $\text{Na}_2\text{S}_2\text{O}_3$ (cm^3) Consumed.			

[LHS NEED TO BE WRITE IN RECOED]

1000 cm³ of 1N Na₂S₂O₃ = 63.54g of copper

Therefore, 1cm³ of 1N Na₂S₂O₃ = 63.54g of copper

Sample 1:

Therefore, (V_{Na₂S₂O₃}) cm³ 0.05 Na₂S₂O₃ = 0.06354 x (V x N)_{Na₂S₂O₃}

i.e. W₄ = g copper

Therefore, percentage of copper in brass sample 1 = $\frac{W_4}{\text{Wt. of brass}} \times 100$

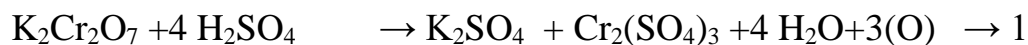
Wt. of brass

[LHS NEED TO BE WRITE IN RECOED]

the solution. Therefore, the end point is marked by the colour change from bluish green to reddish-brown. The end point is sharp only at high acid concentration and therefore, the solution is kept at a very high acidity of H₂SO₄.

A standard solution of FAS is prepared by dissolving a known weight of the salt in a known volume of the solution. A test tube of dil.H₂SO₄ acid is added during dissolution to prevent the hydrolysis of the salt in the solution.

The reaction between Mohr's salt and K₂Cr₂O₇ can be represented as follows:



.....



.....

Equivalent weight of Mohr's salt = molecular weight = 392

Procedure:

(a) Preparation of standard FAS (Mohr's) solution:

Weight accurately given FAS crystals transfer it into a 250 cm³ standard flask. Add a test tube of dilute H₂SO₄ and little distilled water to dissolve the crystals. Make up it to 250 cm³ using distilled water, shake well for uniform concentration.

(b) Determination of COD:

Pipette out 25 cm³ of the given waste sample into conical flask. Add 3/4th test tube of sulphuric acid solution. Then add 10 cm³ of 0.1N K₂Cr₂O₇ solution into the flask. Add 5-6 drops of ferroin indicator and titrate against standard FAS solution till the colour changes from bluish green to reddish brown. Repeat the titration for agreeing values.

Blank: (Blank titrate value (x) will be given)

Blank titration is performed by taking 25 cm³ distilled water in the place of wastewater.

Result:

COD of the given sample of wastewater..... mg/dm³ of oxygen

OBSERVATION AND CALCULATIONS

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

Weight of weighing bottle + FAS crystals = $W_1 = \dots\dots\dots$ g

Weight of empty weighing bottle = $W_2 = \dots\dots\dots$ g

Therefore, weight of FAS (Mohr's salt) = $(W_1 - W_2) = W_3 \dots\dots\dots$ g

$$\begin{aligned} \text{Therefore, molarity of FAS solution} = N_{\text{FAS}} &= \frac{\text{Wt. of FAS taken } (W_3) \times 4}{\text{Eq. Wt. of Mohr's salt } (392)} \\ &= \frac{W_3 \times 4}{392} \end{aligned}$$

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

II estimation of COD value:

Burette: Standard FAS solution.

Conical flask: 25 cm³ wastewater sample + 10 cm³ K₂Cr₂O₇ + 3/4th test tube of H₂SO₄ + 1 gm Ag₂SO₄ + 1 gm HgSO₄ boil, cool

Indicator: Ferroin (1,10 phenanthroline ferrous sulphate)

End point: Bluish green to reddish brown

Burette level	I	II	III
Final burette reading			
Initial burette reading			
Volume of FAS (cm ³) Consumed.			

Volume of FAS consumed to react with 25 cm³ of wastewater y = $\dots\dots\dots$ cm³

[LHS NEED TO BE WRITE IN RECOED]

Given volume of FAS required for blank titration = x =ml

1000 cm³ of 1N FAS = 1 equivalent of oxygen = 8 gm of Oxygen

Therefore, COD value = $\frac{8(x-y) N_{FAS} \times 1000}{V_{\text{wastewater}}}$

$$= \frac{8(x-y) N_{FAS} \times 1000}{25}$$

=

COD value = mg/dm³ of oxygen.

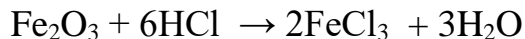
[LHS NEED TO BE WRITE IN RECOED]

5. DETERMINATION OF IRON IN HAEMATITE

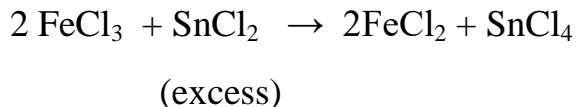
Aim: To determine the percentage of iron in haematite ore.

Principle:

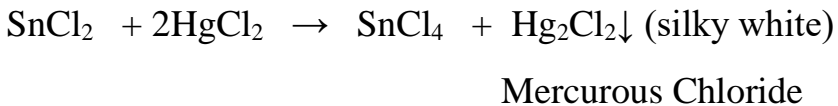
Haematite is an ore of iron, consists of ferric oxide (Fe_2O_3) and small quantities of silica. On treating ore with hydrochloric acid, ferric oxide dissolves in the acid as ferric chloride. Silica is removed by filtration.



Iron in FeCl_3 exists in ferric (Fe^{3+}) and reduced to ferrous (Fe^{2+}) state and then titrated with potassium dichromate solution, which is an oxidizing agent. Ferric chloride (FeCl_3) is reduced by stannous chloride in presence of hydrochloric acid to ferrous chloride (FeCl_2)

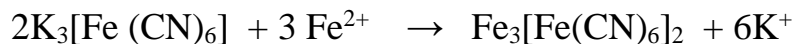


A slight excess of stannous chloride is added to ensure complete reduction of FeCl_3 to FeCl_2 . The excess of SnCl_2 added is destroyed by adding mercuric chloride



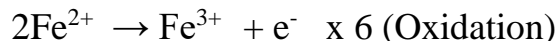
A silky white precipitate of mercurous chloride is obtained. The trial must be discarded if a black precipitate is produced. The trial must also be discarded if no precipitate is obtained because, the complete reduction of ferric ions to ferrous ions is not ensured.

Potassium ferricyanide [$\text{K}_3\text{Fe}(\text{CN})_6$] is used as an external indicator to detect the end point of the titration. It reacts with ferrous ions to form deep blue colored potassium ferric cyanide complex.

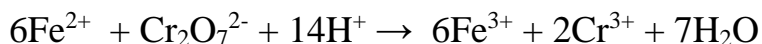


Ferrous ions in the resulting solution is titrated against standard potassium dichromate solution. Blue colour is developed with the indicator droplet as long as ferrous ions exists in the solution. At the end point when all ferrous ions converted into ferric ions, a drop of titrated solution does not develop blue colour with the

indicator droplet. Thus, Ferric ions formed fail to produce blue colour with the indicator droplet.



.....



.....

Procedure:

(a) Preparation of standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution:

Weigh accurately given of $\text{K}_2\text{Cr}_2\text{O}_7$ crystals and transfer it into a 250cm^3 standard volumetric flask. Dissolve the crystals in little distilled water. Make up the solution up to the mark and shake well for uniform concentration.

(b) Estimation of iron:

Pipette out 25cm^3 of given haematite ore solution into a clean conical flask. Add about $1/4^{\text{th}}$ test tube of conc. HCl and heat it to boiling. To the hot solution, add SnCl_2 solution dropwise till the yellow colour is discarded completely. Add two drops in excess. Cool, add 5cm^3 of mercuric chloride solution at a stretch and shake well. A silky white precipitate should be obtained. (if black precipitate or no precipitate is obtained, discard the product). Add two test tubes of distilled water.

Place a number of drops of freshly prepared potassium ferricyanide solution on wax paper. Add a few cm^3 of potassium dichromate solution from the burette into the conical flask and shake well. With the help of a thin glass rod take a drop of the titrated solution and bring it in contact with an indicator droplet. The colour of the indicator drop turns blue. Now add 1cm^3 of $\text{K}_2\text{Cr}_2\text{O}_7$ solution at a time into the conical flask and after each addition of 1cm^3 $\text{K}_2\text{Cr}_2\text{O}_7$ solution, shake the solution well and test the solution drops as before. Repeat this till a drop of the titrated solution does not develop blue colour with the indicator. This titration gives the range of the end point.

Again, pipette out 25cm^3 of the haematite ore solution into a conical flask. Treat with SnCl_2 and HgCl_2 as said before. Add 2 test tube distilled water. This time add most of the potassium dichromate solution required at stretch and further titrate by the dropwise addition of potassium dichromate solution. After the addition of each

drop, test a drop of the titrated solution with the indicator solution drops as explained earlier, till no blue colour is developed with the indicator. Repeat for agreeing values.

Result: Percentage of iron in the given haematite sample is:

OBSERVATION AND CALCULATIONS

I Preparation of standard $K_2Cr_2O_7$ solution:

Weight of weighing bottle + $K_2Cr_2O_7$ crystals = $W_1 = \dots\dots\dots$ g

Weight of empty weighing bottle = $W_2 = \dots\dots\dots$ g

Therefore, weight of $K_2Cr_2O_7 = (W_1 - W_2) = W_3 \dots\dots\dots$ g

Therefore, Normality of $K_2Cr_2O_7$ solution = $N_{K_2Cr_2O_7} =$

$$\begin{aligned} & \frac{\text{Wt. of } K_2Cr_2O_7 \text{ taken} \times 4}{\text{Eq. Wt. of } K_2Cr_2O_7 \text{ salt (49.05)}} \\ & = \frac{W_3 \times 4}{(49.05)} \\ & = \dots\dots\dots N \end{aligned}$$

II Estimation of Iron:

Burette: Standard $K_2Cr_2O_7$ solution.

Conical flask: 25cm^3 of haematite ore solution + $\frac{1}{4}$ test tube conc. HCl + Add SnCl_2 dropwise, till colorless, cool + 5cm^3 of HgCl_2 solution to get silky white ppt. + 2 test tube of water.

Indicator: Potassium ferricyanide [$K_3Fe(CN)_6$] (external indicator)

End point: Failure to develop blue colour with the test droplet.

Burette level	I	II	III
Final burette reading			
Initial burette reading			
Volume of $K_2Cr_2O_7$ (cm^3) Consumed.			

Volume of $K_2Cr_2O_7$ (cm^3) Consumed = $V_{K_2Cr_2O_7} = \dots\dots\dots \text{Cm}^3$

[LHS NEED TO BE WRITE IN RECOED]

Since $N_1V_1 = N_2 V_2$

$$(N \times V)_{K_2Cr_2O_7} = (N \times V)_{\text{ore solution}}$$

$$\text{Therefore, } N_{\text{ore solution}} = \frac{(N \times V)_{K_2Cr_2O_7}}{V_{\text{ore solution}}}$$

$$= \frac{(N \times V)_{K_2Cr_2O_7}}{25}$$

$$= \dots\dots\dots$$

$$N_{\text{ore solution}} = \dots\dots\dots N$$

Wt. of iron/ litre of solution = $W_4 = N_{\text{ore solution}} \times \text{atomic Wt. of iron (55.85)}$

$$= \dots\dots\dots$$

$$W_4 = \dots\dots\dots \text{ g/litre}$$

Given (X).....g of ore solution contains W_4 g of iron.

$$\text{Therefore, 100g of iron contains} = \frac{W_4 \times 100}{X}$$

$$X$$

$$= \dots\dots\dots$$

Therefore, Percentage of iron in the given heamatite ore =

[LHS NEED TO BE WRITE IN RECOED]

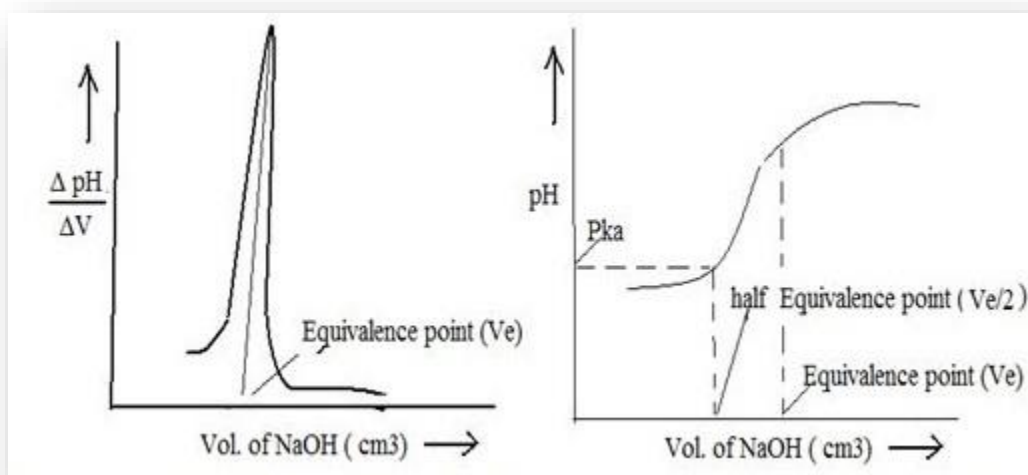
BRIEF PROCEDURE (SYNOPSIS)

Synopsis

PART-A

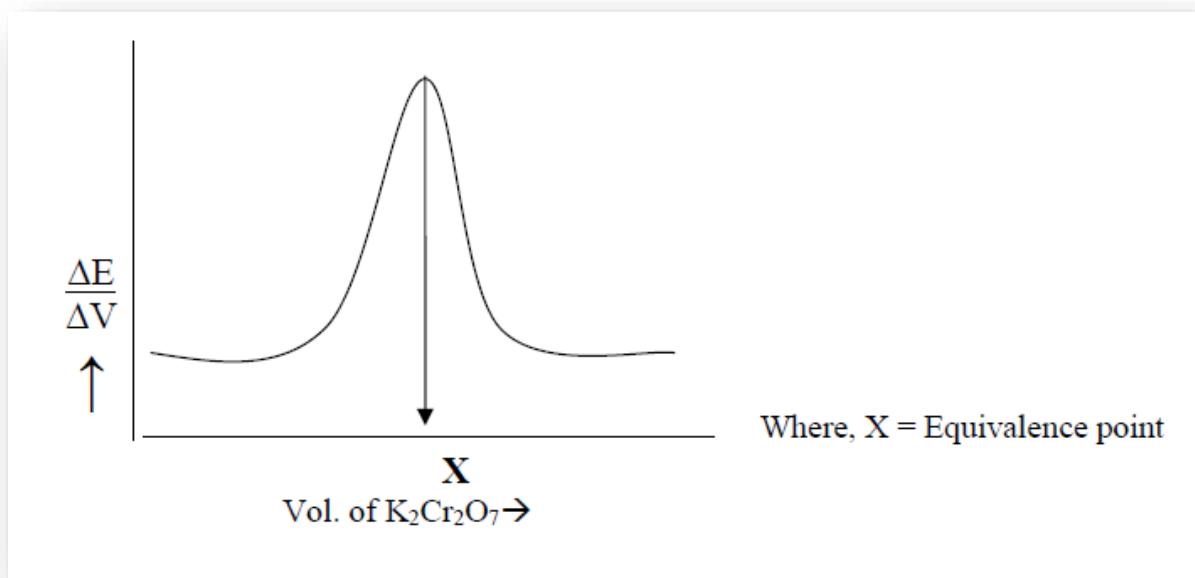
1. DETERMINATION OF pKa VALUE OF WEAK ACID

Pipette out 25 cm³ of the given weak acid into a beaker. Immerse the glass electrode and calomel electrode assembly into it. Connect the electrodes to the pH meter. Add NaOH from the burette in increments of 0.5 cm³ and determine the equivalence point. Plot a graph of pH against volume of NaOH and another graph of $\Delta\text{pH}/\Delta V$ vs volume of NaOH. Using the graphs, determine pKa value of the given weak acid.



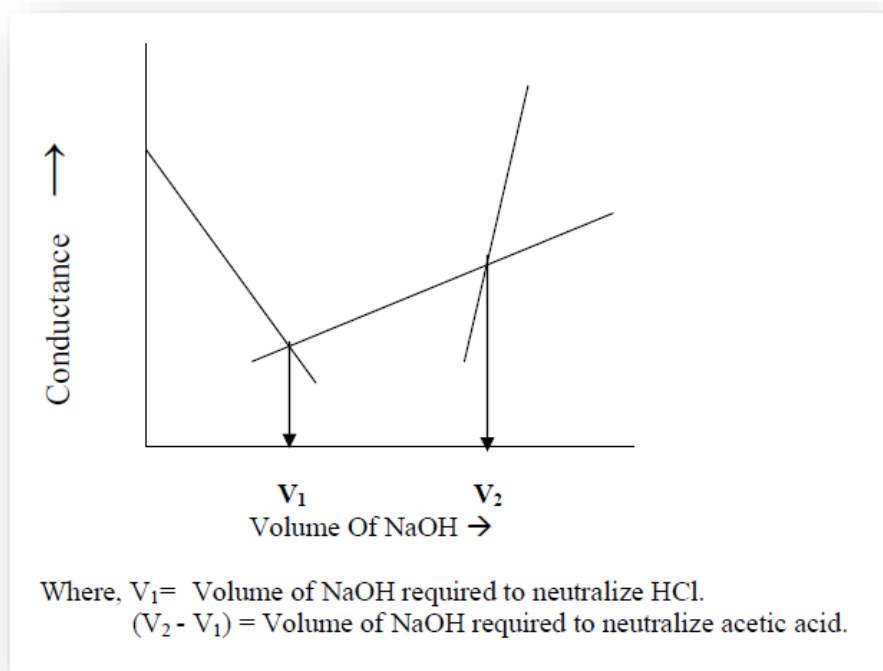
2. ESTIMATION OF FERROUS AMMONIUM SULPHATE USING STANDARD POTASSIUM DOCHROMATE BY POTENTIOMETRIC TITRATION

Pipette out 25 cm³ of FAS into a beaker. Add two test tubes of dilute H₂SO₄. Immerse calomel and platinum electrode assembly into it. Connect the electrodes to a potentiometer and measure the potential. Add K₂Cr₂O₇ from the burette in the increment of 0.5 cm³ and measure the potential after each addition. Plot the graph of $\frac{\Delta E}{\Delta V}$ against volume of K₂Cr₂O₇. Calculate the normality and the weight of FAS in the given solution.



3. CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE USING STANDARD NaOH SOLUTION

Pipette out 25 cm³ of acid mixture into a beaker. Immerse conductivity cell into it. Connect the cell to conductometer and measure the conductance. Add NaOH solution in increment of 0.5 cm³ and measure the conductance after each addition. Plot the graph of conductance against volume of NaOH and determine the equivalence point. Calculate the normality and weight of HCl and CH₃COOH in the given solution.

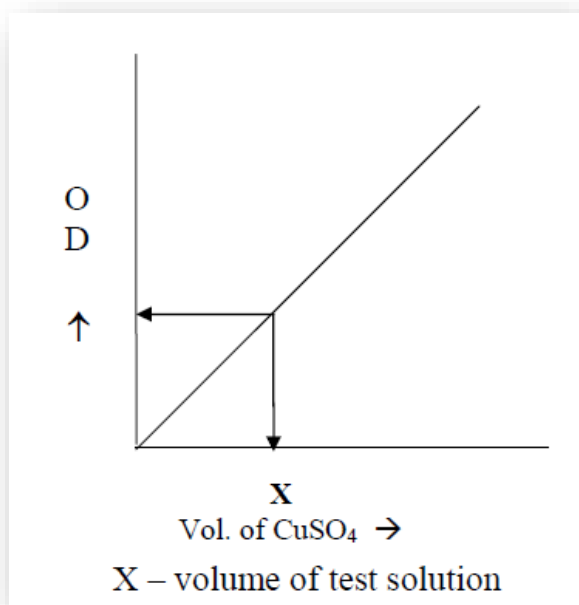


V_1 = Neutralization point of HCl

V_2 = Neutralization point of CH₃COOH

4. COLORIMETRIC ESTIMATION OF COPPER

Transfer 2, 4, 6, 8, 10 cm³ of given copper sulphate solution into separate volumetric flasks. Add 5 cm³ of ammonia to each one of them and also to the given test solution of unknown concentration. Dilute upto the mark using distilled water and mix well. Measure the absorbance of each of this against blank (only ammonia and water) solution. Plot a graph of absorbance (OD) against volume of copper sulphate and determine the concentration of the test solution. λ_{max} for blue color = 620 nm.



5. DETERMINATION OF CO-EFFICIENT OF VISCOSITY OF A LIQUID USING OSTWALD'S VISCOMETER.

Transfer 20 cm³ of the given liquid (organic) into a wider limb of the viscometer and suck the liquid through the narrow limb. Determine the time of flow between two fixed points, one above and another below of the bulb in the narrow limb of viscometer. Repeat and calculate the average time of flow. Pour out the liquid (organic), and wash with acetone and dry it. Now transfer 20 cm³ of water into the wider limb of the viscometer and determine the average time flow for water as before. From the density of the liquid, density of the water and viscosity coefficient of water determine the viscosity of the given liquid (organic).

(t x d) liquid

Viscosity coefficient of the liquid = $\eta_{\text{liquid}} = \frac{\text{-----}}{\text{(t x d) water}} \times \eta_{\text{water}}$ millipoise.

(t x d) water

PART-B

1. 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 cm³ volumetric flask. Add 5 cm³ of ammonia. Dissolve in distilled water and dilute up to the mark, mix well.

Weight of EDTA x 4

Molarity of EDTA = -----

Molecular weight of EDTA (372.24)

Part B: Determination of total hardness

Burette: Standard Na₂EDTA Solution.

Conical Flask: 25 cm³ water sample +5 cm³ buffer (Ammonium hydroxide–ammonium chloride NH₄OH – NH₄Cl) solution.

pH maintained: around 10 (Buffer solution)

Indicator: Eriochrome black-T.

Colour Change: Wine red to clear blue.

Conclusion/Result: From the volume of Na₂EDTA solution consumed, calculate the total hardness of the given water sample.

2. 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 cm³ volumetric flask. Add 5 cm³ of ammonia. Dissolve in distilled water and dilute up to the mark, mix well.

Weight of EDTA taken x 4

Molarity of EDTA = -----

Molecular weight of EDTA (372.24)

Part B: Determination of % of CaO in cement solution

Burette: Standard Na₂EDTA Solution

Conical Flask: 25 cm³ Cement solution + 5 cm³ Diethylamine + 5 cm³ Glycerol +
3/4 test tube of 4N NaOH

pH maintained: around 12.5 (Diethylamine)

Indicator: Patton & Reeder's

Color Change: Wine red to clear blue

Conclusion/Result: From the volume of Na₂EDTA solution consumed, calculate the percentage of CaO in the given Cement solution.

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

Part A: Preparation of Brass sample

Weigh out the given brass sample into a clean beaker. Add 10 to 12 drops of 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 cm^3 volumetric flask and dilute up to the mark with distilled water and mix well.

Part B: Determination of % of Copper in brass

Burette: Standard Sodium thio sulphate solution (Std $\text{Na}_2\text{S}_2\text{O}_3$ solution)

Conical Flask: 25 cm^3 Brass solution + NH_4OH drop wise till bluish White ppt. + dil CH_3COOH till white ppt dissolves & add excess of 3 drops + 1 test tube of 10 % KI.

Indicator: Starch - added towards the end point.

Colour Change: Blue to white ppt.

Conclusion/Result: From the volume of $\text{Na}_2\text{S}_2\text{O}_3$ solution consumed, calculate the percentage of copper in the given brass sample.

4. 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 cm³ volumetric flask. Add 2 test tube of dil H₂SO₄. Dissolve in distilled water and dilute up to the mark, mix well.

Weight of FAS taken X 4

Normality of FAS = -----

Equivalent weight of FAS (392)

Part B: Determination of COD of waste water sample.

Burette: Standard FAS Solution.

Conical Flask: 25 cm³ waste water sample + 10 cm³ K₂Cr₂O₇ + 3/4 test tube of 1:1 H₂SO₄ + 1g of Ag₂SO₄ + 1g of HgSO₄

Indicator: Ferroin (1,10-PHENANTHROLINE IRON(II) SULFATE COMPLEX)

Colour Change: Blush green to reddish brown.

Conclusion/Result: 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.

5. 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 cm³ volumetric flask. Dissolve in distilled water and dilute up to the mark, mix well.

Weight of $K_2Cr_2O_7$ taken X 4

Normality of $K_2Cr_2O_7$ = -----

Equivalent weight of $K_2Cr_2O_7$ (49.09)

Part B: Estimation of Iron in haematite

Burette: Standard $K_2Cr_2O_7$ Solution

Conical Flask: 25 cm³ Haematite ore solution + 1/4th test tube of con. HCl, boil + SnCl₂ drop wise till colorless. Cool & add 1/4th test tube HgCl₂ + 2 test tubes of water

Indicator: Potassium ferricyanide $K_3[Fe(CN)_6]$ External indicator.

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

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

VIVA-VOCE

(Q&A)

VIVA VOCE QUESTIONS

PART-A

1. DETERMINATION OF pKa OF A WEAK ACID USING pH METER

1. How is pKa of a weak acid evaluated from the graph?

pH at half equivalence point gives the pKa of a weak acid

2. What is a weak acid?

Weak acids are weak electrolytes, which ionize incompletely in aqueous solution.

3. Give an example for weak acid?

Acetic acid, Oxalic acid, Formic acid

4. What is pKa of a weak acid?

pKa 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.

5. What is meant by pH?

pH is defined as negative logarithm to base 10 of hydrogen ion concentration.

$pH = -\log_{10}[H^+]$

6. 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.

7. How is the measurement of pH 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 $E = K + 0.0591 \text{ pH}$, where K is a constant.

8. How are pH and pKa related?

According to Henderson-Hasselbalch equation, $pH = pK_a + \log_{10} [\text{salt}] / [\text{acid}]$

9. Why pH at half equivalence point is equal to pKa?

At half equivalence point concentration of salt = concentration of acid, Therefore pH = pKa

10. How are pKa and strength of a weak acid related?

Higher the pKa, lower will be the strength of the weak acid.

11. What are the electrodes used in the measurement of pH and determination of pKa?

Glass electrode (indicator electrode) and calomel electrode (reference electrode).

In this case, glass electrode acts as anode & calomel acts as cathode.

12. 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.

13. What is strong acid?

The substance which ionize almost completely into ions in aqueous solution.

Eg: HCl, H₂SO₄, HNO₃.

14. What is degree of ionization?

$$\text{Degree of ionization} = \frac{\text{No of molecules of the electrolyte which ionizes}}{\text{Total no of molecules of electrolyte}}$$

15. What is Ka?

Ka is the dissociation constant of a weak acid.

$$K_a (\text{CH}_3\text{COOH}) = \frac{[\text{H}_3\text{O}^+] [\text{CH}_3\text{COO}^-]}{[\text{CH}_3\text{COOH}]}$$

2.POTENTIOMETRICTITRATION-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)

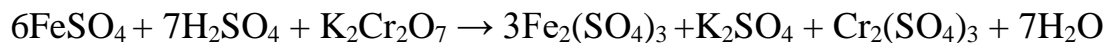
8. What is determining factor in the oxidation-reduction reaction?

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. Ex: Platinum electrode

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 colored 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 colored solutions can be titrated
- ii) Mixture of solutions or very dilute solutions can be titrated.
- iii) The results are more accurate

3.CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE USING STD. NaOH SOLUTION.

1. What is conductivity of an electrolyte solution?

Is a measure of its ability to conduct electricity?

2. What is conductance?

Reciprocal of resistance is called conductance.

3. What is a cell?

A device, which produces an EMF and delivers electric current as the results of a chemical reaction.

4. What factors determine the conductance of a solution?

Two factors determining the conductance of a solution are;

1. Mobility of ions (Higher the mobility, higher is the conductance) and
2. Number of ions (More the number of ions, more is the conductance).

5. 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 .

6. 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?

- i). Accurate for both dilute and concentrated solutions.
- ii). Can be employed for colored solutions.
- iii). Very weak acids such as boric, phenol (cannot be titrated potentiometrically) can be titrated.
- iv). Mixture of acids can be titrated more accurately.

4. 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 colored 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. 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 and path length 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)

$T = I_t / I_0$ 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

$A = \log I_0 / I_t = \log 1/T = -\log T$

I_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?

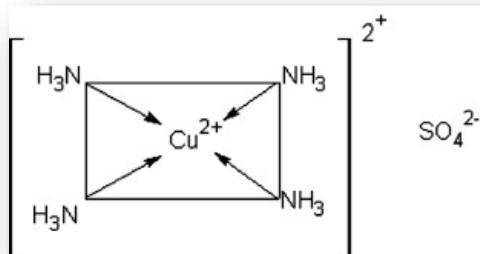
A blank solution is identical in all respect to the test solution except for the absence of test solute.

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

To nullify the absorbance caused due to the coloring 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 coloring 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?

- i) In order to obey Beer-Lambert's law, the solute must not undergo salvation, association, dissociation, hydrolysis or polymerization in the solvent used.
- ii) The colour produced should be sufficiently stable to permit an accurate reading to be taken.
- iii) Clear solutions free from traces of precipitate or foreign substances in either blank or standard test solution should be used.

5. 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 4°C

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?

i) Increase in molecular weight results in an increase in viscosity.

ii) Branched chain compounds have higher viscosity than those involving straight chain compounds.

iii) The polar compounds are more viscous than the non-polar ones. The presence of hydrogen bonds causes the viscosity to increase.

iv) The viscosity of liquids increases by the presence of lyophilic colloids and suspended impurities.

v) 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

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

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.

PART-B

1. ESTIMATION OF TOTAL HARDNESS OF WATER BY EDTA COMPLEXOMETRIC METHOD

1. Why buffer (NH₄OH + NH₄Cl) added?

Buffer 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 HCO₃⁻, SO₄²⁻, Cl⁻, SiO₃²⁻ 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 (HCO₃⁻) of Ca and Mg, while permanent hardness is due to more stable Cl⁻ and SO₄²⁻ 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.

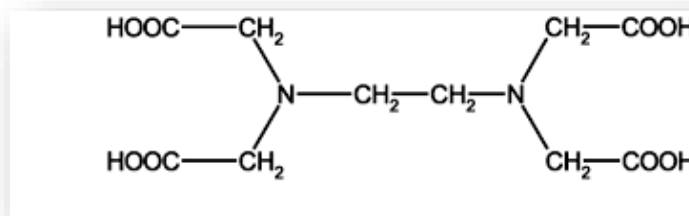
8. How do you express the total hardness of water?

It is expressed as parts per million of CaCO₃.

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.

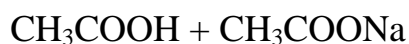
13. Why is ammonia solution added while preparing EDTA solution?

Because to increase the rate of dissolution of EDTA salt.

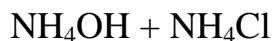
14. What is buffer capacity?

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.

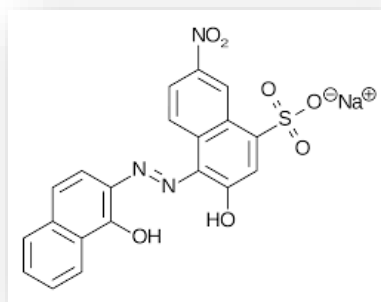
18. What is buffer solution?

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?

Eriochrome black-T

Sodium 4-[(1-hydroxynaphthalen-2-yl-hydrazinylidene)-7-nitro-3-oxo-1-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.



(Stable complex) (protonated)

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.

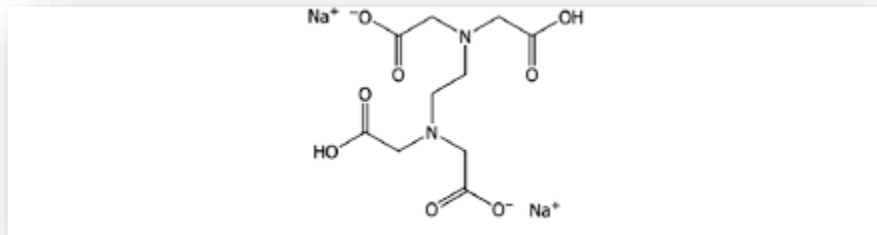
21. Titrations involving EDTA carried out slowly towards the end point. Justify?

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.

23. Structure of disodium EDTA?



2.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?

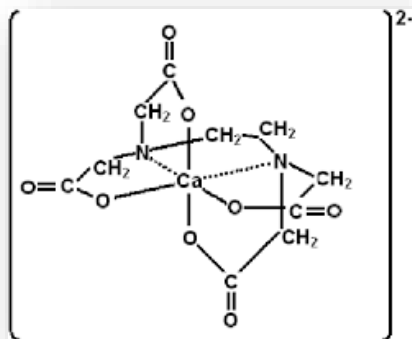
CaO = 50-65%, SiO₂ = 17-25%, Al₂O₃ = 3-8%, Fe₂O₃ = 2-6%, MgO = 0.1 – 5.5%, Na₂O & K₂O = 0.5 – 1.5% and SO₃ = 1-3%

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.



Here Metal is Ca

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?

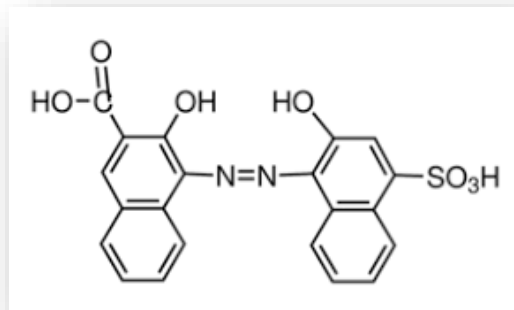
Because it forms a weak complex with calcium ions at pH range of 12-14.

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).



3.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?

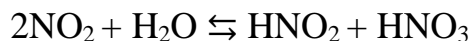
Brass is an alloy of copper (50-90%), zinc (20-40%), small quantities of tin, lead and iron.

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. Its oxide 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 (number) of iodine is liberated is equal to the amount (number) 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.

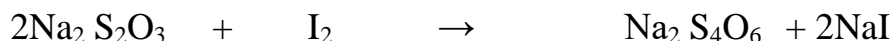
13. Why do you get blue color when starch indicator is added?

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?



sodium thiosulphate

sodium tetrathionate

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

4.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 are 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 oxidizable 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 20⁰C.

6. Why COD greater than BOD value?

Because in COD both organic & inorganic matter get 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?

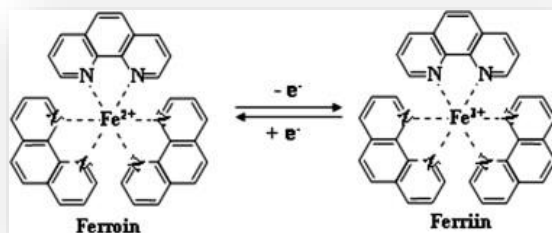
mg/litr of O₂ or mg/dm³ of O₂ or ppm of O₂.

10. Name the oxidizing agent?

Potassium di chromate.

11. Explain the colour change during the titration?

Ferroun indicator is red in colour in the reduced form with the composition, [Fe(O-Phen)₃]²⁺ (ferroun), When ferroun is added to conical flask containing waste water & potassium dichromate. The indicator gets oxidized by potassium dichromate (oxidizing agent) to form [Fe(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 Ferroun which is in red colour which is the end point of titration.



Ferroun Red in color

Ferrin Bluish green color

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 solvents. 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. What are the products formed after COD analysis?

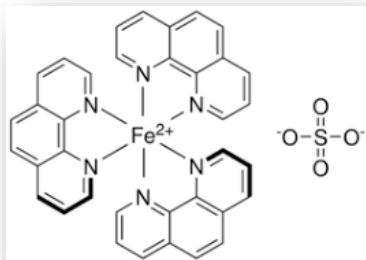
During COD analysis organic matter is completely oxidized to carbon dioxide and water by acidified potassium dichromate solution.

18. 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.

19. What is the composition of ferroin?

Ferroin is ferrous 1, 10-Phenanthroline sulphate.



20. 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.

21. 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.

22. What is the limitation of COD?

One of the chief limitations of COD test is inability to differentiate between biologically oxidizable 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 exist in nature.

5. 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 (Fe_2O_3)

4. Give the other forms of iron ore?

Magnetite (Fe_3O_4), Siderite (FeCO_3), Iron pyrite (FeS_2)

5. Why HCl is added?

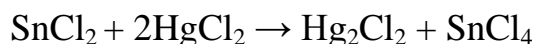
HCl is added to convert Fe_2O_3 to FeCl_3 , as Fe_2O_3 does not react with $\text{K}_2\text{Cr}_2\text{O}_7$

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?

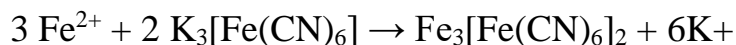
$\text{K}_3[\text{Fe}(\text{CN})_6]$, Potassium ferri cyanide as external indicator.

10. What is the color of $\text{K}_3[\text{Fe}(\text{CN})_6]$ with ferrous ion?

Potassium ferri cyanide produces an intense deep blue color with ferrous ions due to the formation of ferriferrocyanide complex.

11. 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.



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

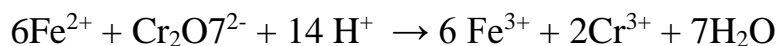
Potassium Ferro cyanide cannot be used as an indicator because Ferro cyanide does not react with ferrous ion.

13. 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.

14. 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.



15. Can Fe be determined using internal indicators?

Yes, Fe can be determined using internal indicators.

16. Name the internal indicator used for iron determination?

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

17. What is the oxidation state of Fe in ore?

Fe^{+3}

GENERAL VIVA VOCE QUESTIONS

1. Define molarity?

It is the number of moles of solute dissolved in 1 liter of the solution.

2. Define normality?

It is the number of gram equivalents of the solute dissolved in 1 liter of the solution

3. Define Equivalent weight?

Equivalent weight of substance = Molecular mass of the substance / X

X = Basicity of an acid, Acidity of a base, valency of salt, oxidation state of an oxidizing / reducing agent or change in oxidation state.

For example,

X = Acid

Equivalent weight = Molecular mass of the substance / basicity

Basicity is the maximum no. of H⁺ ions liberated on dissociation

Eg: HCl = 1; Equivalent weight = 36.5/1 = 36.5

H₂SO₄ = 2; Equivalent weight = 98/2 = 49

X = Base

Equivalent weight = Molecular mass of the substance / Acidity

Acidity is the maximum no. of OH⁻ ions liberated on dissociation

Eg: NaOH = 1; Equivalent weight = 40/1 = 40

X = Salt

Equivalent weight = Molecular mass of the substance / valency

Eg: Na₂CO₃; which dissociates to give Na²⁺ and CO₃²⁻. It means 2 cations and 2 anions. So, valency for Na₂CO₃ is 2.

X = oxidizing / reducing agent

Equivalent weight = Molecular mass of the substance / change in oxidation state

Eg: KMnO_4 in acidic medium the oxidation state (O.S) changes +7 to +2. So, change in O.S is 5.

So, Equivalent weight = $158/5 = 31.6$

4. Define Molality?

It is the number of gram equivalents of the solute dissolved in 1kg of the solution

5. Define Indicator?

Indicator are the chemical substance which indicates the completion of the reaction by changes its color.

6. Define Standard solution?

A solution of known concentration is called standard solution. A known amount of solute in known amount of solvent is also said to be STD solution.

7. What are the types of Indicators?

There are three types of Indicators.

Internal: Phenolphthalein, starch, EBT, Patton and Reeder's.

External: Potassium ferricyanide.

Self: Potassium permanganate.

Acid base: Phenolphthalein

Metal ion: Eriochromo black-T, Patton and Reeder's

Redox: Ferroin.

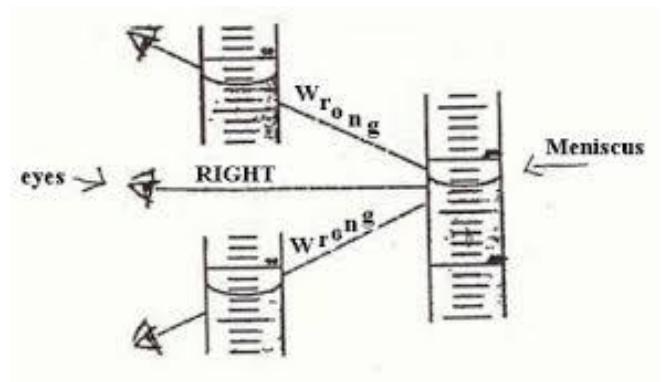
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General Instructions for Volumetric Analysis



BURETTE KNOB ADJUSTMENT

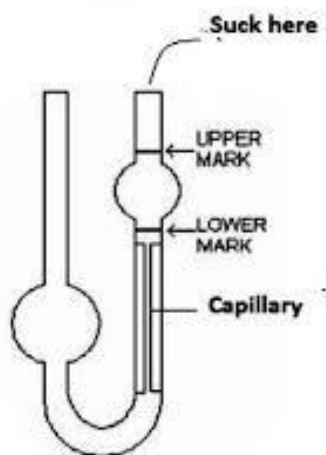


MINISCUS OBSERVATION

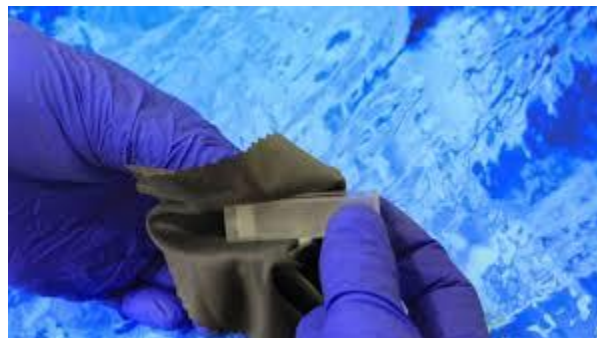


TITRATION

General Instructions for Instrumentation Analysis



VISCOMETER



**CUVETTE WIPING FOR
COLORIMETER READING**



**CONDUCTOMETER – NOTE DOWN THE
READING AFTER STABILIZATION**

