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"The difference between a successful person and others is not a lack of strength, not a lack of knowledge, but rather in a lack of will."

~Vincent T. Lombardi

Instrumental Experiments





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Record of observation:

1. Laboratory temperature =⁰C
2. Density of water = d_w = g/cc
3. Viscosity co-efficient of water = η_w = mpoise
4. Density of given liquid = d_l = g/cc

Tabulation: Flow Time Measurement

Liquid	Time of Flow in “seconds”
Given liquid (t_l)	1.
	2.
	3.
	Mean time t_l =s
Water (t_w)	1.
	2.
	3.
	Mean time t_w =s



Title of the Experiment:

Determination of Viscosity co-efficient of a given liquid using Ostwald's Viscometer

Experiment No. : 1

Date :

Principle:

Viscosity of a liquid may be defined as the resistance that one part of a fluid offers to the flow of another part of the liquid. Viscosity is produced by the shearing effect of moving one layer of the fluid past another. It may be thought as caused by the internal friction of the molecule themselves. When a liquid is in laminar flow through a tube the layer close to the surface of the tube is almost stationary and the layer at the axis of the tube moves faster than any other layer. A slow moving layer exerts a friction on its nearest layer. The coefficient of viscosity (η) is defined as the force per unit area required to move a layer of fluid with a unit velocity difference past another parallel layer at unit distance away. In cgs system of units, the coefficient of a fluid is expressed in poises.

The viscosity coefficient of a liquid is given by **Poiseuille's equation**.

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

where v is the volume of the liquid of viscosity coefficient (η) which flows in time t through a capillary tube of radius r and length l under a pressure head of P .

We know that, $P = h d g$, where, h = height, d = density, g = acceleration due to gravity.

If equal volumes of two liquids are allowed to flow through the same capillary under identical conditions,

$$\frac{\eta_l}{\eta_w} = \frac{\pi h d_l g r^4 t_l}{8 v l} \times \frac{8 v l}{\pi h d_w g r^4 t_w} = \frac{d_l t_l}{d_w t_w}$$

The flow times for the liquid and water are determined in Ostwalds viscometer. Knowing the densities of the liquid and water and also knowing the viscosity coefficient of water, viscosity coefficient of liquid can be calculated. Since viscosity is dependent on temperature the measurement can be carried out in water bath to reduce the change in temperature.



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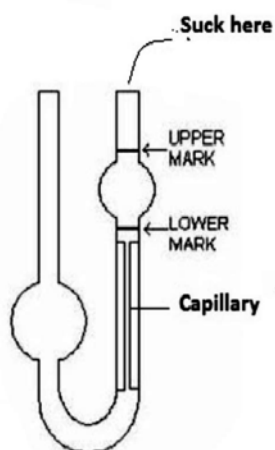
Calculation:

$$\text{Viscosity coefficient of the given liquid, } \eta_l = \frac{d_l \times t_l}{d_w \times t_w} \times \eta_w$$

=

=

= m poise at°C



Ostwald's Viscometer



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Procedure:

Take a clean and dry viscometer and fix the viscometer vertically to a stand. Using a burette transfer a known volume (say 10 ml of the lower bulb) of water through wider limb. Suck the water above the upper mark of the viscometer. Allow it to flow freely through the capillary, when the level of the water just crosses upper mark, start the stop clock and when the water just crosses the lower mark, stop the stop clock. Note down the time of flow in seconds (t_w). Repeat the same procedure twice.

Pour out the water, rinse the viscometer with acetone and dry it. Repeat the experiment by taking exactly the same volume (as that of water) of the given liquid whose viscosity is to be determined and record the time of the flow in seconds (t_l). Repeat the procedure to get agreeing values.

Result: Viscosity coefficient of the given liquid is.....m poise at°C

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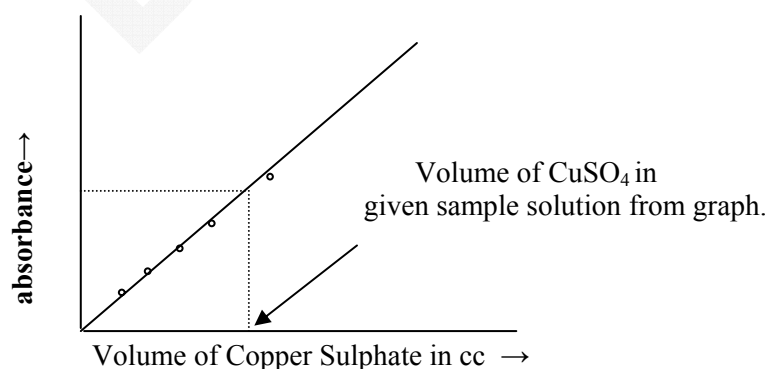
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Tabulation:

Filter with wavelength _____ is selected as it shows maximum Optical Density for Cu^{2+} ions.

Flask Number	Volume of CuSO_4 in cc 'V'	Optical density	Weight of CuSO_4 ($5 \times V$) mg	Weight of Copper ($1.2724 \times V$) mg
Blank	0			
1	5			
2	10			
3	15			
4	20			
5 (Test solution)	(Ux)			

Nature of Graph:





Title of the Experiment:

Colorimetric estimation of Copper

Experiment No. : 2

Date :

Principle

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

$$I_0 = I_a + I_r + I_t$$

For a glass-air interface I_r is negligible, therefore

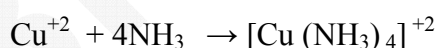
$$I_0 = I_a + I_t$$

$I_t/I_0 = T$ called the Transmittance, $\log I/T = \log I_0/I_t$ is called the absorbance or optical density. The relation between absorbance, A , concentration c (expressed in mole/dm³) and path length, t (expressed in cm) is given by Beer- Lambert's law.

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

where ϵ is the molar extinction coefficient, t is the path length and is constant for a given substance at a given wavelength. If t , is the path length and is kept constant, then $A \propto C$. Hence a plot of absorbance against concentration gives a straight line.

A series of standard solution of copper salt is treated with ammonia to get blue cuprammonium complex and is diluted to a definite volume.



deep-blue complex

The absorbance of each of these solutions is measured at 620 nm since the complex shows maximum absorbance at this wavelength. The absorbance values are plotted against concentration to get a calibration curve.

A known volume of the test solution is treated with strong ammonia and diluted to the same volume as above. The absorbance of this solution at 620 nm is measured and its concentration is determined from the calibration curve.

Procedure :

- 1) Clean six 100 ml capacity volumetric flasks are taken and named as 1, 2, 3, 4, 5 (blank) and 6 (unknown).
- 2) A series of standard solutions of different concentrations of cupric ions are prepared by



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Calculation:

a. 1000 cc of CuSO_4 solution contains = 5 g of CuSO_4

1 cc of CuSO_4 solution contains = 5 mg of CuSO_4

b. 249.68 g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ = 63.54 g of Cu

5 (i.e., x) mg of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ contains = $\frac{x \times 63.54}{249.68}$ mg of Cu

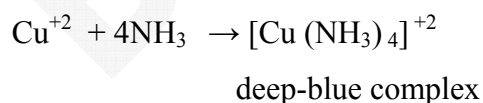
= $5 \times 0.2544 = 1.2724$ mg of Cu.



adding given CuSO_4 solution from burette 5, 10, 15, and 20cc respectively into 1st, 2nd, 3rd, and 4th volumetric flask.

- 3) Now add constant volume (5 cc) of liquid ammonia (NH_4OH solution) to all 6 volumetric flasks.
- 4) The solution is diluted up to the mark with distilled water for all volumetric flasks.
- 5) Put the stopper and shake it well to get the homogeneous solution.
- 6) The absorbance of each of the solution is measured at 620 nm, since the complex shows maximum absorbance at this wavelength.
- 7) Tabulate the readings. Plot a graph of optical density versus volume of copper sulphate solution. From the calibration curve the volume of the unknown solution is determined i.e. the volume of the test solution and calculate the amount of copper in the given solution.
- 8) This is done as follows,
Select the filter 620 nm and place the glass cell containing blank solution in the colorimeter. Adjust the knob to zero optical density (OD)
- 9) Then take the glass cell containing 1st complex solution, place it in the colorimeter and note down the OD. Similarly find the optical densities of other solutions of different concentrations also.
- 10) Finally find the OD of the unknown solution and plot the graph.

Note: The deep blue complex observed is the Cuprammonium complex.



Result:

1. From the graph, the volume of copper sulphate in the test solution is = cc
2. The weight of copper in the given test solution is = mg

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Record of Observation:

Solution in Microburette : Std. NaOH solution.

Solution in Beaker : Given acid mixture

Tabulation:

[illegible]



Title of the Experiment

Conductometric estimation of an Acid mixture using standard NaOH solution

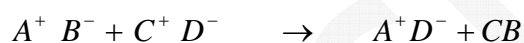
Experiment No. : 3

Date :

Principle:

The determination of equivalence point of a titration by this method is based upon the measurement of the conductance during the course of titration, which varies in different manner before and after the equivalence point. This is due to the reason that electrical conductance of a solution depends upon the number of ions present and their ionic mobilities. Thus, on plotting the conductance against the volume of the titrant added we get two branches of straight lines; the point of intersection of the lines gives the equivalence point.

Suppose a solution of an electrolyte AB is titrated against a solution of another electrolyte CD, and the following reaction takes place.



If during the titration there is no appreciable volume change and one of the product, say CB, is either slightly ionized or insoluble, then addition of CD will result in a decrease or increase of the conductance of the solution, depending upon the relative mobility of B^- and D^- (B^- is being replaced by D^-). After the equivalence point the conductance changes in different manner depending upon the mobility of C^+ .

Let us consider the titration of Acid mixture with NaOH.

When a mixture containing acetic acid and hydrochloric acid, is titrated against an alkali, strong acid (HCl) will be neutralized first. The neutralization of the weak acid (CH_3COOH) commences after the complete neutralization of strong acid. Thus, the conductance titration curve will be marked by two breaks: the first one corresponds to the equivalence point of HCl and the second to that of CH_3COOH .

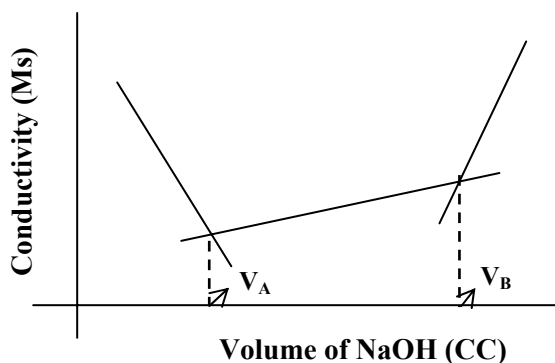
Let V_1 and V_2 be the volume of alkali corresponding to first and second breaks respectively. Then

$V_1 \text{ cm}^3$ of NaOH required to neutralise HCl

$(V_2 - V_1) \text{ cm}^3$ of NaOH required to neutralise CH_3COOH



Nature of Graph:



Calculation:

Volume of NaOH required to neutralize HCl = V_A cc (from graph) =

$$\text{Normality of HCl} = \frac{\text{Normality of NaOH} \times V_A}{\text{Total volume of acid}}$$

=

$$= 'X' \text{ N} =$$

Strength of HCl = Normality of HCl x Eq. Wt. of HCl

$$= 'X' \text{ N} \times 36.5$$

=

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

Volume of NaOH required to neutralize CH_3COOH = $V_{(B-A)}$ cc (from graph) =

$$\text{Normality of } \text{CH}_3\text{COOH} = \frac{\text{Normality of NaOH} \times V_{(B-A)}}{\text{Total volume of acid}}$$

=

$$= 'Y' \text{ N} =$$

Strength of CH_3COOH = Normality of CH_3COOH x Eq. Wt. of CH_3COOH

$$= 'Y' \text{ N} \times 60$$

=

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



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Procedure:

After calibrating the Conductivity meter, a clean beaker containing the given acid mixture solution is taken.

Immerse the conductivity cell in a acid mixture solution. Record the initial conductivity of the solution. Fill the micro burette with the Std. NaOH solution. From the micro burette add Std. NaOH solution 0.5 cc at a time to the mixture. Stir or blow air well into the solution and note-down the conductivity after each addition. The conductivity initially decreases, then rises slowly and then rises sharply. Continue the addition of base and measure the concentration until the three limbs of graph are traced fully.

Plot the conductivity value against the corresponding volume of NaOH added. The nature of the graph is as shown in fig. 1. The point A corresponds to the neutralization of the strong acid; hence the volume of NaOH corresponding to point A is the volume required for the neutralization of HCl (V_A). Similarly, the point B corresponds to the neutralization of all acids. Hence, volume corresponding to (B-A) gives the NaOH required to neutralize the weak acid i.e. $V_{(B-A)}$.

Results : i) Normality of HCl is.....N & Strength of HCl is.....g/lit

ii) Normality of CH_3COOH is.....N & Strength of CH_3COOH is.....g/lit

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Title of the Experiment:

Potentiometric estimation of FAS using standard $K_2Cr_2O_7$ solution

Experiment No. : 4

Date :

Principle: A Potentiometric titration may be defined as a titration in which the end point is detected by measuring the change in potential of a suitable electrode (which responds to the change in concentration) during the titration. The electrode which responds to the change in concentration of the ion is called the indicator electrode. The indicator electrode is combined with a reference electrode (whose potential does not change during the titration) to form a cell and the e.m.f of the cell so formed is measured during titration. The e.m.f. of the cell changes gradually till the end point and changes rapidly at very close to the end point. After the end point the change is once again gradual. When e.m.f. E is plotted as ordinate and the volume of titrant V added as abscissa, the point of inflection of the curve corresponds to the equivalence point or the end point of the titration.

A better method is by plotting the first derivative curve, in which $\Delta E / \Delta V$ is plotted against V and the end point is obtained at the maximum of the curve.

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



The presence of oxidised 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 Pt wire. Thus when the titration is commenced, both Fe^{2+} and Fe^{3+} 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 potential is given by,

$$E = E^\circ + \frac{2.303 RT}{F} \log \frac{[Fe^{3+}]}{[Fe^{2+}]}$$

It is combined with a reference electrode (calomel if potentiometer and glass if P^H meter is used) and the e.m.f. of the cell is measured. 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 $[Fe^{3+}] / [Fe^{2+}]$ 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 converted completely into Fe^{3+} by $K_2Cr_2O_7$, the electrode $(Pt/ Fe^{3+}, Fe^{2+})$

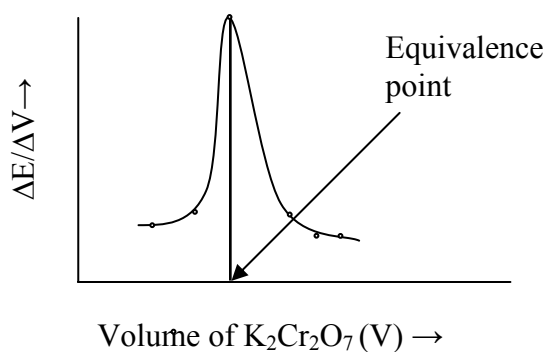


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Nature of Graph:

$\Delta E/\Delta V$ Vs Volume of $K_2Cr_2O_7$



Calculation:

$$\text{Normality of FAS solution} = \frac{\text{Normality of } K_2Cr_2O_7 \times \text{Equivalence point (from graph)}}{\text{Volume of FAS taken}}$$

=

$$= 'X' \text{ N} =$$

$$\text{Amount of FAS in the given solution} = 'X' \times \text{Equivalent weight of FAS}$$

$$= 'X' \times 392$$

=

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



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ceases to exist. But the presence of slight excess of $K_2Cr_2O_7$ brings in the existence of $(Pt/ Cr^{6+}, Cr^{3+})$ electrode. Thus after the end point it is the potential of the $(Pt/ Cr^{6+}, Cr^{3+})$ which is going to change. Because of both the factors, i.e., increase in the value of $[Fe^{3+}] / [Fe^{2+}]$ and change of electrode from $(Pt/Fe^{3+}, Fe^{2+})$ to $(Pt /Cr^{6+}, Cr^{3+})$ there is a large change in potential at the end point.

Procedure:

After standardizing the potentiometer, the supplied Ferrous Ammonium Sulphate solution into a clean beaker is taken. Add 1 test tube of dil. Sulphuric acid to it. Immerse the Platinum and Calomel electrodes into the solution. Note down the potential of the FAS solution.

Fill the microburette with standard potassium dichromate ($K_2Cr_2O_7$) solution. Titrate the FAS solution against standard $K_2Cr_2O_7$ by adding 0.5 cc of $K_2Cr_2O_7$ solution each time. Stir well and note-down the EMF after each addition. Continue the titration until the EMF remains almost constant.

Determine the equivalence point by differential method i.e. by plotting a graph of $\Delta E/\Delta V$ against volume of potassium dichromate solution. The equivalence point is the volume of potassium dichromate at the maxima. From this calculate the normality of the FAS solution & hence the amount of FAS present in the solution.

Result: Amount of FAS in the given solution is.....g/lit.

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Record of observations:

- 1) Solution in burette ----- Std.NaOH solution
- 2) Solution in beaker ----- given weak acid

Tabulation:

Volume of NaOH added V cc	pH	ΔpH	ΔV	$\Delta pH / \Delta V$
		----	----	----



Title of the Experiment:

Determination of pK_a of a weak acid using pH Meter

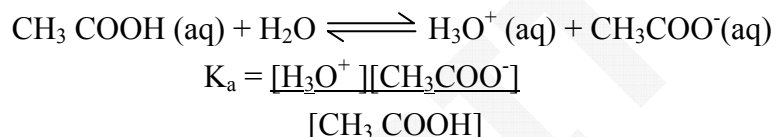
Experiment No. : 5

Date :

Principle :

The strength of acid is experimentally measured by determining its equilibrium constant or dissociation constant (K_a). Since strong acids are strong electrolytes, they are ionized almost completely in aqueous solutions. It is not meaningful to study the ionic equilibrium of strong acid and calculate their equilibrium constants as the unionized form is present to such a small extent. Hence the study of equilibrium and calculation of K_a is applicable only to weak acids.

Acetic acid ionizes feebly as,



p^{K_a} is a modern method of expressing acid strengths. p^{K_a} = -log₁₀ K_a

p^{K_a} is determined by measuring the changes in **p^H** of acid solution at different amounts of the base added. Higher the value of **p^{K_a}**, weaker is the acid. [Eg. Formic acid : 3.75, acetic acid : 4.76, propionic acid : 4.88, chloroacetic acid: 2.86, bromo acetic acid :2.90, Oxalic acid : K₁ = 1.27 and K₂ = 4.27].

During the titration of an acid with a base the **p^H** of the solution rises gradually at first, then more rapidly and until at the equivalent point, there is a very sharp increase in **p^H** for a slight addition of base. Once past the equivalence point, the **p^H** increases only slightly on addition of excess base. The titration curve is obtained by plotting changes in **p^H** at different amounts of the base added and the equivalence point is determined. Once the titration is started, the solution contains weak acid and also its salt with strong base. Thus the mixture is a buffer mixture. The **p^H** of such a mixture is given by **Henderson – Hasselbalch** equation. According to equation,

$$\text{p}^{\text{H}} = \text{p}^{\text{K}_a} + \log_{10} [\text{salt}] / [\text{acid}]$$

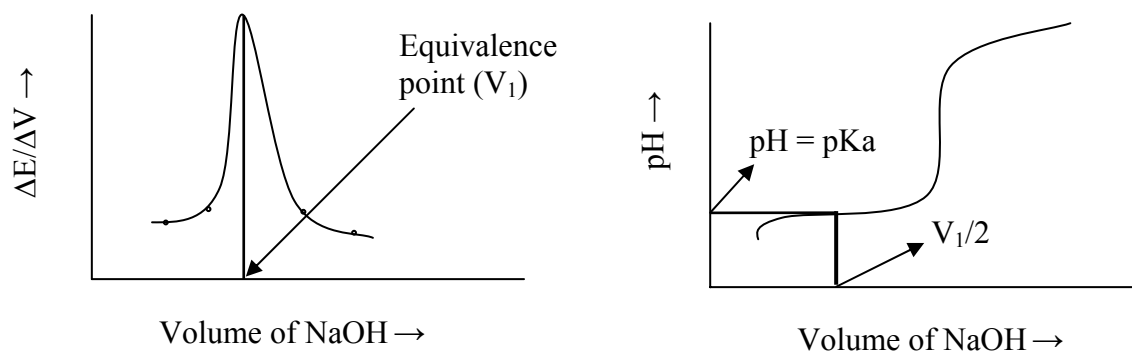
At half equivalence point, 50% of acid is converted to salt and so [salt] = [acid] and therefore, **p^H** at half equivalence point gives the **p^{K_a}** of weak acid.



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Nature of Graph:





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Procedure:

After standardizing the pH meter, the given weak acid (acetic acid or formic acid) is taken in a clean beaker. Immerse glass-electrode and Calomel electrode or combined glass electrode into the weak acid solution and note down the pH of the solution. From the micro burette add Std. NaOH solution 0.5 cc at a time to the acetic acid. Stir well and note-down the pH after each addition till there is an abrupt increase in the pH. Continue the titration and note down the pH readings for four more readings after the jump in pH value. Plot a graph of $\Delta\text{pH} / \Delta V$ against volume of NaOH added and note the equivalence point. From the graph of pH vs volume of NaOH added find out pH at half-equivalence point, which gives the pKa of the weak acid.

Results: From graph – I Equivalence point (V_1) = -----

From graph – II pH at half equivalence point ($V_1 / 2$) = pKa = -----

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Tabulation:

Standards and sample	Observed Concentration in ppm	
	Sodium	Potassium
Standard 1 (Na 40 ppm & K 40 ppm)		
Standard 2 (Na 70 ppm & K 70 ppm)		
Sample 1		
Sample 2		



Title of the Experiment:

Flame Photometric estimation of Sodium and Potassium in the given sample of Water

Experiment No. : 6

Date :

Principle:

Emission of characteristic radiation by element and the correlation of the emission intensity with the concentration of the element is the basis of flame photometry.

When a solution containing the sample element or the ion is aspirated into the flame, a series of changes take place at the flame.

First, the solvent gets evaporated leaving behind the salt in the flame

1. The salt then gets evaporated into vapours of the salt, which further undergo disassociation into the constituent atoms
2. Metal atoms formed in the flame absorb heat energy from the flame and get electronically excited into their higher energy level
3. Excited metal atoms fall back to their ground state by emitting the energy in the form of radiations
4. The intensity of the light radiation emitted is proportional to the no. of atoms in the excited state which in turn is proportional to the no. of atoms in the flame are the concentration of the solution fed into the flame
5. Thus the concentration of the solution is related to the intensity of emitted radiation
6. By measuring the intensity of the emitted radiation by a flame photometer concentration can be determined.

Procedure:

- i) Prepare the stock and standard solutions of sodium and potassium as follows
 - 1.a A stock solution of sodium (1000 ppm) is made by dissolving 0.635 g of AR grade NaCl in 250 cc of double distilled water.
 1. b A stock solution of potassium (1000 ppm) is made by dissolving 0.477 g of AR grade KCl in 250 cc of double distilled water.

With the help of stock solutions prepared in step 1) make the standard solution of the element under analysis having 40 ppm and 100 ppm concentrations using double distilled water for



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standardization of the flame photometer. A mixed standard for sodium and potassium should be used.

- 1.c** Dilute stock solutions 1:100 to get 1 mg Na/100 cc and 1 mg K/100 cc, which are equivalent to 10 ppm
- i) Start the electrical supply and switch on the air supply. Stabilize the air. This needle should be steady at the mark.
 - ii) Switch on the gas and maintain the gas fuel mixture so that the blue flame is seen through the viewing window.
 - iii) Refer the manufacturer's manual to calibrate the flame photometer by using set of standard solutions.
 - vii) Determine concentration of Na and K (ppm) in the sample after the calibration.

Results: Concentration of Na in the given water sample 1 and 2 &.....mg/L (ppm)

Concentration of K in the given water sample 1 and 2 &.....mg/L (ppm)

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"When you can do the common things of life in an uncommon way, you will command the attention of the world."

~George Washington Carver

Volumetric Experiments





Record of observations:

Part A: Preparation of std.solution of disodium salt of EDTA

Weight of bottle + disodium salt of EDTA = 'W₁' g =

Weight of bottle = 'W₂' g =

Weight of disodium salt of EDTA = 'W' g = (W₂ - W₁) =

Molarity of disodium salt of EDTA = $\frac{W \times 4}{372.24 \text{ (Mol.Wt.of EDTA)}} = \frac{\quad \times 4}{372.24}$

= 'X' M =

Part B: Titration: Determination of total hardness of water sample

Solution in Burette : Std. disodium salt of EDTA solution.

Solution in Conical flask : 25cc hard water sample + 2cc Buffer solution.

Indicator used : Eriochrome Black -T

Colour change : Wine red to clear blue

Tabulation:

Burette levels	I	II	III	Mean BR
Final level				
Initial level				
Difference				
				cc



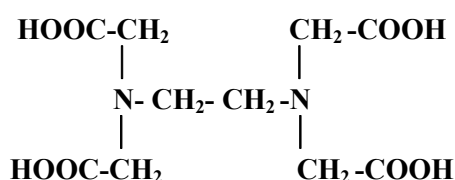
Title of the Experiment:

Determination of Total Hardness of a sample of Water using Disodium salt of EDTA

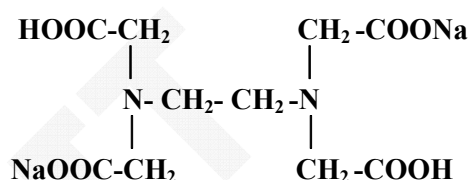
Experiment No. : 7

Date :

Principle: Hardness of water is due to the presence of calcium and magnesium salts in water. Ethylenediamine-tetraacetic acid (EDTA) forms complexes with a large number of cations including Ca^{2+} and Mg^{2+} ions. Therefore the hardness of water may be determined by titrating a known volume of water sample with standard solution of EDTA, using Erichrome Black-T as an indicator. Accordingly, it is possible to determine the total hardness of water using EDTA reagent.



EDTA (H_4Y)



$\text{Na}_2\text{H}_2\text{Y}$

The EDTA molecule (H_4Y) has two easily replaceable hydrogen atoms and the resulting ion after ionisation may be represented as H_2Y^{2-} . The latter forms complexes with metal ions as follows.



where M^{2+} is Ca^{2+} and Mg^{2+} present in water. Reaction (1) can be carried out quantitatively at a p^{H} of 10. Since the reaction involves the liberation of H^+ ions, a buffer mixture has to be used to maintain a p^{H} of 10. The buffer mixture used in titration is $\text{NH}_3\text{-NH}_4\text{Cl}$. The hardness of water is usually measured as parts per million of CaCO_3 . Since EDTA is sparingly soluble, its di-sodium salt, $\text{Na}_2\text{H}_2\text{Y}$ is used for preparing the reagent.

Procedure:

Part A: Preparation of standard disodium salt of EDTA solution:

Weigh out the weighing bottle containing ---- g of disodium salt of EDTA using an electronic balance. Transfer the crystals carefully onto a funnel placed over 250 cc volumetric flask and then find the weight of empty weighing bottle and note down. Pour a minimum quantity of deionised water and allow the crystals to flow it into a 250 cc volumetric flask. Remove the funnel. Add 5cc of 1:1 NH_3 to improve the solubility of EDTA. Dissolve the crystals by



Calculation:

$$(M_1 V_1)_{\text{HARD WATER}} = (M_2 V_2)_{\text{EDTA}}$$

$$M_{\text{1of HARDWATER}} = \frac{(M_2 V_2)_{\text{EDTA}}}{V_{\text{1of HARDWATER}} (25\text{cc})}$$

=

= 'y' =

$$\text{Wt / litre of CaCO}_3 \text{ equivalent hardness} = M_{\text{HARD WATER}} (y) \times \text{Mol. wt. Of CaCO}_3 (100)$$

=

= 'z' = g

$$\therefore 10^6 \text{cc (1 million cc) of hard water sample contains} = \frac{z \times 10^6}{1000} \text{ g of CaCO}_3$$

=

=

$$\text{Thus, Total Hardness of the given water sample} = \text{mg/ litre OR ppm of CaCO}_3$$



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Swirling the flask gently. Dilute the solution up to the mark with deionised water; stopper the flask and mix the solution thoroughly by inverting the flask several times so that a homogeneous solution results. Calculate the molarity of the EDTA solution.

Part B: Titration: Determination of total hardness of water sample:

Pipette out 25cc of the given hard water sample into a clean conical flask. Add about 2cc of $\text{NH}_3\text{-NH}_4\text{Cl}$ buffer solution and 2-3 drops of EBT indicator. Titrate this weak wine red coloured complex against standard EDTA solution until colour changes from wine red to clear blue. Note down burette reading and repeat the titration.

Result: Total Hardness of given water sample ismg/litre OR ppm

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Record of Observations:

Part A: Preparation of std. solution of disodium salt of EDTA

Weight of bottle + disodium salt of EDTA = 'W₁' g =

Weight of bottle = 'W₂' g =

Weight of disodium salt of EDTA = 'W₃' g = (W₂ - W₁) =

Molarity of EDTA = $\frac{W_3 \times 4}{372.24 \text{ (Mol. Wt. of EDTA)}} = \frac{\quad \times 4}{372.24}$

= 'X' M =

Weight of the Cement in the given solution = 'W' g =

Part B: Titration: Determination of total hardness of water sample

Solution in Burette : Std. EDTA solution.

Solution in conical flask : 25cc of Cement solution + 10cc of 4N NaOH solution +
5cc of diethyl amine + 5cc of Glycerol.

Indicator used. : Patton & Reeder's indicator.

Colour change : Turbid Wine-red to clear blue.

Tabulation:

Burette levels	I	II	III	Mean BR
Final level				cc
Initial level				
Difference				



Title of the Experiment:

Determination of CaO in the given sample of Cement by Rapid EDTA method

Experiment No. : 8

Date :

Principle:

Cement contains silicates of iron, aluminium and calcium. Calcium oxide is a prime constituent of cement. The general composition of Portland cement is given below:

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

In the estimation of calcium in cement, the given cement sample is treated with concentrated hydrochloric acid. The insoluble silica is filtered off and the filtrate which contains calcium ions is titrated at p^H of 12-14 against EDTA using Patton and Reeder's indicator. The latter is used because, Eriochrome Black T forms very weak complex with calcium ions. Diethylamine is added to maintain a p^H of about 12.5 and glycerol is added to get a sharp end point. This method facilitates the determination of Ca²⁺ ions although Mg²⁺ ions are present in the cement solution (Mg²⁺ do not interfere at this condition)

Procedure:

Part A: Preparation of Standard EDTA solution:

Weigh out accurately g of disodium salt of EDTA and transfer it into a 250 cc volumetric flask through a funnel. Dissolve the crystals in deionised water and add 5cc of 1:1 NH₃ and then make it up to the mark using deionised water. Mix thoroughly.

Part B: Titration: Estimation of % of CaO

Rinse and fill the burette with std. disodium salt of EDTA solution. Now pipette out 25cc of given Cement solution into a clean conical flask. Add 5cc of 1:1 Glycerol and shake the solution. Add 5 cc of diethylamine to adjust pH of the solution at 12. Add 10 cc of 4N NaOH to precipitate Mg²⁺ ions as Mg(OH)₂ to avoid the interference of Mg²⁺ ions in the titration. Shake it well. Now add 2-3 drops of Patton & Reeder's indicator and titrate against std. EDTA solution till the colour changes from wine-red to clear blue. Note down the burette reading and repeat the procedure to get concordant readings.

Results: The percentage of CaO in the supplied sample of Cement solution is%

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Calculation:

$$(M_1 V_1)_{\text{CEMENT SOLN.}} = (M_2 V_2)_{\text{EDTA}}$$

$$M_{\text{1of CEMENT SOLN.}} = \frac{(M_2 V_2)_{\text{EDTA}}}{V_{\text{1of CEMENT SOLN.}} (25\text{cc})}$$

=

$$= 'y' =$$

$$\text{Wt / litre of CaO present} = M_{\text{CEMENT SOLN.}} (y) \times \text{Mol. wt. Of CaO (56)}$$

=

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

$$\text{Percentage of CaO present in the supplied sample of cement solution} = \frac{'z' \times 100}{W}$$

=

$$= \dots\dots\dots \%$$



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Record of observations:

Part A: Preparation of std. brass solution

Weight of Brass piece = 'W' g =

Brass Solution: Known weight of Brass sample + 1 test tube of Nitric Acid + 1 g of urea. Boil, cool and add ammonium hydroxide drop wise till a deep bluish white precipitate appears. Add 3/4 th test tube of acetic acid till light blue colour appears. Transfer the solution into the volumetric flask and make it upto 250cc using deionised water.

Part B: Titration: Estimation of Copper in Brass

- Solution in Burette : Standard Sodium thiosulphate solution.
- Solution in conical flask : 25 cc Brass solution + 2 test tube of 10% KI solution.
- Indicator used : 2cc of freshly prepared Starch solution.(to be added near end point when solution turns pale yellow)
- Colour change : Disappearance of blue colour to milky white colour.

Tabulation:

Burette levels	I	II	III	Mean BR
Final level				cc
Initial level				
Difference				



Title of the Experiment:

Determination of Percentage of Copper in Brass using standard Sodium thiosulphate solution

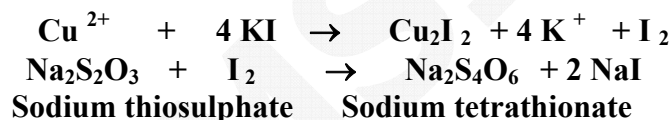
Experiment No. : 9

Date :

Principle:

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

A solution of brass is made by dissolving the sample in minimum amount of nitric acid. Urea is added to expel the oxides of nitrogen. Nitric acid (strong oxidising agent) is neutralised by the addition of ammonium hydroxide. Acetic acid is added to make the solution faintly acidic. When potassium iodide is added to this ore solution containing cupric ions, it oxidises iodide to iodine. The iodine liberated is titrated against sodium thiosulphate using starch as indicator. The volume of sodium thiosulphate consumed is a measure of amount of copper present.



Procedure:

Part A: Preparation of Brass solution:

Weigh out accurately the supplied Brass sample and transfer it into a clean 250 cc beaker. Add a quarter test tube of 1:1 nitric acid in each. Cover the mouth of the flasks with the glass funnel and boil till the alloy dissolves completely. Add 10 cc of deionised water and gently boil till all the brown coloured oxides of nitrogen are expelled completely. Add 1.0 g of urea and continue boiling for some more time to expel all the fumes. Cool and add one more test tube of ion exchange water. Add dilute ammonium hydroxide drop wise until a dark blue colour results (Initially a slight precipitate blue is formed which dissolves in excess ammonium hydroxide). Acidify this solution with dilute acetic acid to get back the light blue colour. Transfer the solution carefully to a 250 cc std. flask. Wash the beaker and the funnel 2-3 times with deionised water and transfer the washings to the std. flask and make it up to the mark.



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Calculation:

$$(NV)_{\text{BRASS.SOLN.}} = (NV) \text{ Na}_2\text{S}_2\text{O}_3$$

$$N_{\text{BRASS.SOLN}} = \frac{(NV) \text{ Na}_2\text{S}_2\text{O}_3}{V_{\text{BRASS.SOLN}}} =$$

$$\text{Wt of Cu /250cc of brass soln.} = \frac{N_{\text{BRASS}} \times \text{Eq. Wt of Cu (63.54)}}{4}$$

4

=

$$= (a) = \quad \text{g}$$

$$\text{Percentage of Cu in the given brass sample} = \frac{a \times 100}{W}$$

W

=

= %



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Part B : Estimation of Copper

Pipette out 25 cc of the brass solution to a conical flask. Add 2 test tube full of 10 % KI solution. Titrate the iodine liberated against standard sodium thiosulphate solution taken in the burette immediately till the mixture turns pale yellow. Add 2 cc of starch indicator and continue the titration till the blue colour disappears and a white precipitate is left behind. Note the volume of thiosulphate solution added. Repeat the titration for concordant values.

Note:

- 1.The brass solution should not contain mineral acids as they would oxidise Iodine to Iodic acid and also decompose the sodium thiosulphate(hence the solution is neutralised with ammonia solution and acidified with acetic acid).
2. Starch is added near the end point when the concentration of iodine is low.If added earlier(at high iodine concentrations), a water insoluble starch – iodine complex is formed which makes detection of the endpoint difficult.

Result: The percentage of Copper in the given Brass sample.....%

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Record of Observations:

Part A: Preparation of std. solution of potassium dichromate

Weight of bottle + $K_2Cr_2O_7$ crystals = ' W_1 ' g =

Weight of empty bottle = ' W_2 ' g =

Weight of $K_2Cr_2O_7$ crystals = ' W_3 ' g = ($W_2 - W_1$) =

Normality of $K_2Cr_2O_7$ = $\frac{W_3 \times 4}{49 \text{ (Eq. Wt. of } K_2Cr_2O_7\text{)}} = \frac{\quad \times 4}{49}$
= ' X ' N =

Weight of Haematite Ore = ' W ' g =

Part B: Estimation of % of Iron in Haematite

Solution in Burette : Std. $K_2Cr_2O_7$ solution.

Solution in Conical flask : 25cc of Ore solution + $\frac{1}{4}$ th tt of conc. HCl ---Heat + $SnCl_2$ drop wise till yellow colour disappears----cool + $\frac{1}{4}$ th tt $HgCl_2$ till silky white ppt.

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

Colour change : No change in the colour of the indicator when a drop of reaction mixture is brought in contact with it.

Tabulation:

Burette levels	I	II	III	Mean BR
Final level				cc
Initial level				
Difference				



Title of the Experiment:

Determination of Iron in the given sample of Haematite ore solution using Potassium dichromate Crystals by external indicator method

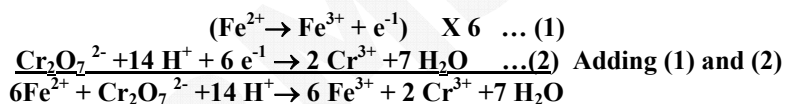
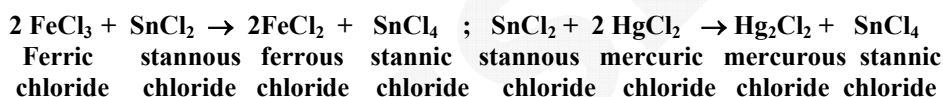
Experiment No. : 10

Date :

Principle:

Haematite is an important ore of iron containing mainly Fe_2O_3 and a small amount of silica (SiO_2). A known weight of the ore is digested with Conc.HCl and the insoluble residue mainly containing silica is removed by filtration. The filtrate is used for estimation of iron.

Iron is present in the solution as Fe^{3+} (ferric) ions. It is reduced to Fe^{2+} (ferrous) ions using stannous chloride (SnCl_2) in hot condition in the presence of Conc.HCl. The excess of stannous chloride added is oxidised by treating the solution with mercuric chloride. The ferrous ions in the resulting solution is titrated against std. Potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) using potassium ferricyanide [$\text{K}_3\text{Fe}(\text{CN})_6$] as external indicator. Potassium ferricyanide produces an intense deep blue colour with ferrous ions due to the formation of ferriferrocyanide complex.



Procedure:

Part A: Preparation of Std. $\text{K}_2\text{Cr}_2\text{O}_7$ solution:

Weight out accuratelyg of $\text{K}_2\text{Cr}_2\text{O}_7$ crystals and transfer it into a 250 cc volumetric flask and make it up to the mark using deionised water. Mix well.

Part B: Titration: Estimation of Iron in Haematite ore solution

Pipette out 25 cc of the given haematite ore solution into a clean conical flask. Add $\frac{1}{4}$ tt of conc. HCl and heat the solution to boiling. Add SnCl_2 solution to the hot yellow ore solution drop wise till the yellow colour just disappears. Cool the solution and then add HgCl_2 solution in a lot. (If precipitate is black reject the sample). A white silky precipitate of mercurous chloride Hg_2Cl_2 is formed. Place a number of drops of external indicator on a wax



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Calculation:

$$(NV)_{\text{Fe- solution}} = (NV)_{\text{K}_2\text{Cr}_2\text{O}_7 \text{ soln.}}$$

$$N_{\text{Fe- solution}} = \frac{(NV)_{\text{K}_2\text{Cr}_2\text{O}_7 \text{ soln.}}}{V_{\text{Fe- solution}}}$$

=

=

$$\text{Wt of Fe /litre of ore soln.} = N_{\text{Fe- solution}} \times \text{Eq. Wt of Fe (55.85)}$$

=

$$= (z) = \dots\dots\dots \text{ g}$$

$$\text{Percentage of iron present in the given sample of Haematite ore} = \frac{'z' \times 100}{W}$$

=

$$= \dots\dots\dots \%$$



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paper with the help of clean glass rod. Now titrate the solution with potassium dichromate solution, at definite interval withdraw a drop of the solution from conical flask with glass rod and mix it with one of the drops of indicator on the wax paper. Appearance of dark blue/green colour indicates that end point has not reached. Continue the titration by adding 1 cc each time and repeat the process of withdrawing a drop of solution from conical flask & mixing it with indicator drop on the wax paper. When the drop of the reaction mixture fails to give any colour change with the indicator drop, it indicates the end point. Note down the burette reading.

Note: Clean the glass rod after every test. Repeat the titration by taking another 25 cc of the haematite solution. This time add most of the potassium dichromate solution required at a stretch and then titrate drop wise just before 1 cc of before the end point observed onwards from previous reading. Mix the contents of the flasks after every addition and test a drop of the indicator as described above till the colour of the indicator drop does not change. Repeat for agreeing values.

Remember: 1) SnCl_2 should be added dropwise to the hot solution. 2) Mercuric chloride should be added rapidly in cold condition. Formation of grey/black precipitate on the addition of mercuric chloride indicates that excess SnCl_2 has been added in which case trial has to be discarded. Also if no precipitate is observed discard the solution 3) Excess SnCl_2 will give Hg (grey ppt.) 4) potassium ferricyanide forms a blue ppt with Fe (II), hence when the titrated solution just fails to produce a colour with the indicator, the titration has to be stopped.

Result: The percentage of Iron in the supplied sample of Haematite Ore is.....%

Staff Signature



Record of Observations:

Part A: Preparation of standard solution of ferrous ammonium sulphate (Mohrs Salt)

1. Weight of bottle + FAS crystals = g
 2. Weight of empty bottle = g
 3. Weight of FAS crystals = g
- Normality of FAS = $\frac{\text{weight of FAS} \times 4}{\text{Eq. wt. of FAS}} = \frac{\text{.....} \times 4}{392}$
= 'Z' N =

Part B: Back titration:

Solution in Burette : Std. FAS solution.
 Solution in Conical flask : 25cc (V cc) waste water sample + 5 cc $\text{K}_2\text{Cr}_2\text{O}_7$ + 3/2 tt of H_2SO_4
 Indicator Used : 2 drops of Ferroin solution.
 Colour change : Greenish-blue to Brownish-red colour.

Tabulation:

Burette levels	I	II	III	
Final level			 'V ₁ ' cc
Initial level				
Difference				

Volume of FAS used for back titration = (V₁) cc
 (i.e., the volume of FAS consumed by unreacted $\text{K}_2\text{Cr}_2\text{O}_7$ solution)

Part C: Blank titration:

Solution in Burette : Std. FAS solution.
 Solution in Conical flask : 25cc distilled water sample + 5 cc $\text{K}_2\text{Cr}_2\text{O}_7$ + 3/2 tt of H_2SO_4
 Indicator Used : 2 drops of Ferroin solution.
 Colour change : Greenish-blue to Brownish-red colour.

Tabulation:

Burette levels	I	
Final level	 'V ₂ ' cc
Initial level		
Difference		

Volume of FAS used for blank titration = (V₂) cc
 (i.e., the volume of FAS consumed by 5cc $\text{K}_2\text{Cr}_2\text{O}_7$ solution)



Title of the Experiment:

Determination of COD of the given Industrial Waste water sample

Experiment No. : 11

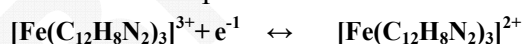
Date :

Principle:

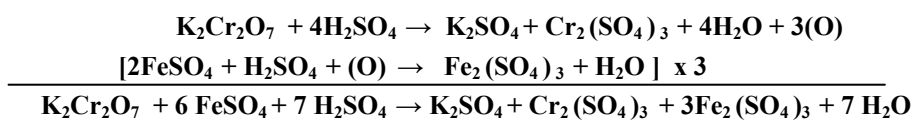
Chemical oxygen demand (COD) parameter is a measure of the total oxidisable impurities present in the sewage. This includes both biologically oxidisable and biologically inert but chemically oxidisable impurities in the water. It is the amount of oxygen equivalent used while oxidizing the chemically oxidisable impurities present in water, with a strong chemical oxidant, potassium dichromate in acid medium. It is expressed in mg.dm^{-3} or ppm.

A known volume of the waste water sample is refluxed with excess of dichromate solution in sulphuric acid medium and in the presence of Ag_2SO_4 and HgSO_4 . Waste water contains organic impurities like straight chain aliphatic compounds, aromatic hydrocarbons, straight chain alcohols, acids, pyridine and other oxidisable impurities. Potassium dichromate oxidizes all oxidisable impurities. Silver sulphate catalyses the oxidation of straight chain organic compounds, aromatics and pyridine but Cl^- ions precipitate silver ions as silver chloride in the absence of HgSO_4 . HgSO_4 is added to avoid interference of Cl^- ions by forming soluble complex with them. The amount of potassium dichromate solution consumed corresponds to the COD of the water sample. To calculate the COD, a blank titration without the waste water sample is carried out.

Ferriin (1,10 phenanthroline-iron (II) complex) is used as the indicator in the titration to detect the end point. The indicator is intensely red in colour. Strong oxidizing agent oxidizes the indicator to iron (III) complex which has a pale blue colour.



As long as dichromate solution is present in the solution, the indicator is in the oxidized form, imparting blue colour to the solution. The solution becomes blue green as titration continues due to the formation of Cr (III) ions as result of reduction of $\text{Cr}_2\text{O}_7^{2-}$ ions. When dichromate is completely exhausted in the solution, the oxidized form of the indicator gets reduced to the reduced form, imparting red brown colour to the solution. At the end point the colour changes from blue green to red brown. The end point is sharp only at high acidic concentration. A standard solution of Mohr's salt is prepared by dissolving a known weight of the salt in a known volume of the solution. A test tube of dilute sulphuric acid is added during dissolution to prevent the hydrolysis of the salt solution. The reaction between Mohr's salt and potassium dichromate can be represented as follows.



Equivalent weight of Mohr's salt = Molecular weight = 392.



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Calculation:

Volume of FAS required for Back titration = 'V₁' cc=

Volume of FAS required for Blank titration = 'V₂' cc=

Volume of FAS equivalent to the oxidisable impurities = (V₂- V₁) cc.

Normality of FAS = 'Z' N=

1000cc of 1N FAS solution = 1 equivalent of oxygen = 8 g of oxygen

1 cc of 1 N FAS = 8 mg of oxygen

Thus, Chemical Oxygen Demand (COD) of the given waste water sample,

$$= \frac{(V_2 - V_1) \times 'Z' N \times 8 \times 1000}{V (25)}$$

=

=mg/lit or ppm of oxygen



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Procedure:

Part A: Preparation of standard Mohrs salt solution.

Weigh accurately g of Mohrs salt, transfer into a volumetric flask, dissolve it in a little deionised water, add a test tube of dilute sulphuric acid and then make it up to the mark with deionised water.

Part B: Determination of COD:

Back Titration:

Pipette out 25cc of waste water sample into a clean conical flask. Then pipette out 5 cc of $K_2Cr_2O_7$ solution into the flask. Add 3/2 tt of dil. H_2SO_4 . Add 2-3 drops of Ferroin indicator and titrate against standard Ferrous Ammonium Sulphate solution until the solution turns from greenish-blue to brownish-red colour. Note the burette reading as 'Y'cc.

Blank Titration:

Repeat the above procedure by using 25cc of deionised water instead of waste water sample. Note the burette reading as 'X'cc.

Result: Chemical Oxygen Demand (COD) of the given waste water sample is..... mg/L

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Record of observations:

Solution in Burette : Std. H_2SO_4 solution
Solution in Conical flask : 25 ml given water sample
Indicators used : Phenolphthalein and methyl orange indicator.
Colour change : i) Pink to colourless
 ii) Yellow to orange red

Tabulation:

Burette levels	I	II	III	Mean BR
Initial level				
Phenolphthalein end point				A=.....cc
Methyl orange end point				B=.....cc

Calculation:

Normality of H_2SO_4 = (Normality of stock solution x Volume given) / 250

=

Volume of sample titrated = V_{cc} =cc

Volume of H_2SO_4 used to P-end point = A cc =cc

Volume of H_2SO_4 used to M-end point = B cc =cc

Phenolphthalein Alkalinity = $\frac{N \times A \times 1000 \times 50}{V}$ =

=mg/lit in terms Of CaCO_3

Methyl orange Alkalinity = $\frac{N \times B \times 1000 \times 50}{V}$ =

=mg/lit in terms Of CaCO_3



Title of the Experiment:

Determination of Total Alkalinity of a given Water Sample using standard Sulphuric acid

Experiment No. : 12

Date :

Principle:

Alkalinity refers to the capability to neutralise acid. Alkalinity of water is due to the presence of OH^- (hydroxide), HCO_3^- (bicarbonate) and CO_3^{2-} (carbonate) ions. The presence of these ions can be determined by titrating against standard acid solution.

In the presence of acid, alkaline causing ions in water undergo the following reactions.

- a) $\text{OH}^- + \text{H}^+ \rightarrow \text{H}_2\text{O}$
- b) $\text{CO}_3^{2-} + \text{H}^+ \rightarrow \text{HCO}_3^-$
- c) $\text{HCO}_3^- + \text{H}^+ \rightarrow \text{H}_2\text{CO}_3$

It's an acid-base titration, where the presence of strong bases can be determined by using phenolphthalein as the indicator and weak bases by using methyl orange as an indicator, i.e., phenolphthalein end point corresponds to the completion of 1 and 2 reactions where as methyl orange end point corresponds to completion of reaction 3. Therefore total alkalinity can be determined by titrating the water sample against a strong acid solution using both phenolphthalein and methyl orange as an indicator.

END POINT: pink to colour less (phenolphthalein), yellow to red (methyl orange)

Procedure:

Pipette out 25ml of the water sample into a conical flask. Add 4-5 drops of phenolphthalein indicator and titrate this sample against Std. H_2SO_4 until the pink colour caused by phenolphthalein just disappears. Note down this reading as phenolphthalein end point (A cc). Now add 3-4 drops of methyl orange in the same solution. It will give light yellow color. Continue the titration against the same H_2SO_4 until the light yellow color changes to orange red. Now note the total volume of acid as B cc. This is methyl orange end point. The same procedure is repeated till the concordant readings are obtained.

Result: The total alkalinity of the given water sample is.....mg/L

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"The only place where success comes before work is in the dictionary."

~ Vidal Sassoon



Viva-voce Questions





Determination of Viscosity co-efficient 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 ps = 1 kg m⁻¹ 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?

- 1) Increase in molecular weight results in an increase in viscosity.
- 2) Branched chain compounds have higher viscosity than those involving straight chain compounds.
- 3) The polar compounds are more viscous than the non polar ones. The presence of hydrogen bonds cause the viscosity to increase.
- 4) The viscosity of liquids increases by the presence of lyophilic colloids and suspended impurities.
- 5) 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

$$\eta = \frac{\pi Pr^4 t}{8vl}$$

where, P = hgd.



v = volume of the liquid, r = radius of the tube, l = length of the tube, P = 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

Colorimetric estimation of Copper

1. What is the basic principle of colorimetric measurements?

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

2. What is colorimetry?

Chemical analysis through measurements of absorption of light radiation in the visible region of the spectrum (400-760nm) with respect to a known concentration of the substance is known as colorimetry.

3. What forms the basis for colorimetric determination?

The variation of colour of a system with change in concentration of some component forms the basis for the colorimetric determination

4. What is photoelectric colorimeter?

It is an electrical instrument, which measures the amount of light absorbed using a photocell.



5. What are filters? Why are they used?

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

6. What is wavelength?

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

7. What is wave number?

It is reciprocal of wavelength. Wave number = $1/\lambda$ = Frequency / Velocity of light

8. What is frequency?

It is the number of waves passing through a point per second. It is represented by ν

9. State Beer's law.

The intensity of a beam of monochromatic light decrease exponentially as the concentration of the absorbing substance increases arithmetically.

10. State Lambert's law.

When a monochromatic light passes through a transparent medium, the rate of decrease in intensity with the thickness of the medium is proportional to the intensity of the light. or The intensity of the emitted light decreases exponentially as the thickness of the absorbing medium increases arithmetically.

11. State Beer-Lambert's law.

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

$$A = \log I_0 / I_t = \epsilon Cl;$$

(ϵ = Molar extinction coefficient, c = concentration, l = path length)

Therefore, $A \propto Cl$

12. What is calibration curve?

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

13. What is meant by transmittance?

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

$$T = I_t / I_0$$

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

14. What is absorbance or optical density?

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

15. How is optical density 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. What is blank solution?

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

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

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

20. 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.

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

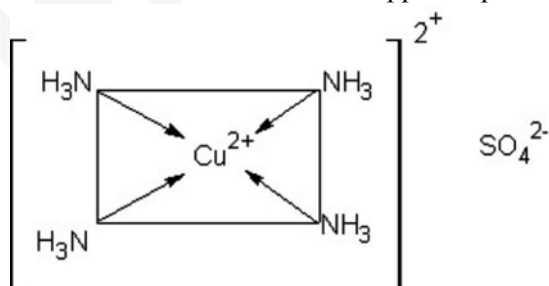
Different volumes of solutions are taken to prepare standard solutions of different concentration, which are used to plot a calibration curve.

22. Mention a few advantages of photoelectric colourimetric determinations.

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

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

23. 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

24. Why is ammonia added? Why is that same amount of ammonia added?

Ammonia is added to get cuprammonium sulphate $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4$ a dark blue complex. Same amount of ammonia is added to nullify the absorbance due to any colouring impurities present in ammonia.

25. 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.



26. Mention a few important criteria for a satisfactory colorimetric analysis.

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

Conductometric estimation of an Acid mixture using standard 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;

- Mobility of ions (Higher the mobility, higher is the conductance) and
- 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.

- Specific conductance.
- Equivalent conductance
- 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 + CH_3COOH 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 slightly due to the formation of CH_3COONa , a strong electrolyte during neutralization of CH_3COOH . Further there is a rapid increase in the graph, because of the presence of free OH^- ions (Conductivity : $198\text{ Ohm}^{-1}\text{m}^{-1}$).

16. 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.

17. What are the advantages of conductometric titration over visual potentiometric titrations?

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

Potentiometric estimation of FAS using standard $\text{K}_2\text{Cr}_2\text{O}_7$ 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.

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

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?

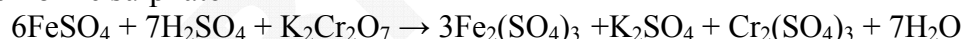
The determining factor is the ratio of the concentrations of the oxidized and reduced forms.

9. What is indicator electrode?

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

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

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



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

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

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

Due to formation of green coloured chromic sulphate.

13. Why emf rapidly change at the equivalence point?

At the equivalence point, emf depends on concentration of both ferric ion and dichromate ion. Therefore emf rapidly change at the equivalence point.

14. What are the advantages of potentiometric titrations?

- i) Turbid, fluorescent, opaque or coloured solutions can be titrated
- ii) Mixture of solutions or very dilute solutions can be titrated.
- iii) The results are more accurate



Determination of pKa of a weak acid using pH Meter

1. How is p^{Ka} 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

4. What is p^{Ka} of a weak acid?

pKa is defined as, negative logarithm to the base 10 of dissociation constant of weak acid.

i.e., $pKa = -\log_{10} Ka$, Where Ka is the dissociation constant of the weak acid.

5. What is meant by p^H ?

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

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

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 p^H made?

The measurement of pH is made by determining the emf of the cell containing glass electrode and the calomel electrode immersed in the test solution. The emf of the cell is expressed by the equation $E = K + 0.0591 \text{ pH}$, where K is a constant.

8. How are p^H and p^{Ka} related?

According to Henderson-Hasselbalch equation, $p^H = p^{Ka} + \log_{10} [\text{salt}] / [\text{acid}]$

9. Why pH at half equivalence point is equal to p^{Ka} ?

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

$$p^H = p^{Ka}.$$

10. How are p^{Ka} 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 p^{Ka} ?

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.

After which the concentration of hydroxyl ions increases due to further addition of NaOH.

So pH increases suddenly.



Determination of Total Hardness of a sample of Water using Disodium salt of EDTA

1. Why is ammonia ammonium chloride buffer added?

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

2. What is hard water?

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

3. What is total hardness of water?

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

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

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

5. How is hardness of water caused?

Hardness of water caused by divalent metallic cations in association with anions such as HCO_3^- , SO_4^{2-} , Cl^- , SiO_2 etc. The principal hardness causing cations are calcium and magnesium.

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

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

7. How temporary hardness is be removed?

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

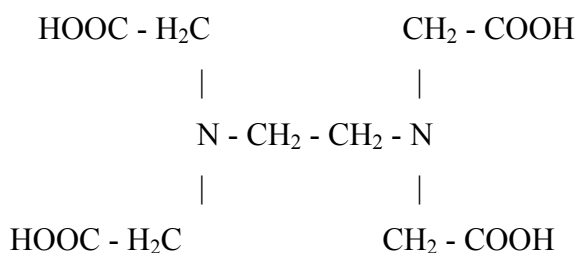
8. How do you express the total hardness of water?

It is expressed as parts per million of $CaCO_3$.

9. What is EDTA?

Ethylene diamine tetra acetic acid.

10. Write a structural formula of EDTA.





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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?

$\text{CH}_3\text{COOH} + \text{CH}_3\text{COONa}$

16. Give an example of basic buffer?

$\text{NH}_4\text{OH} + \text{NH}_4\text{Cl}$

17. What is reserve alkalinity?

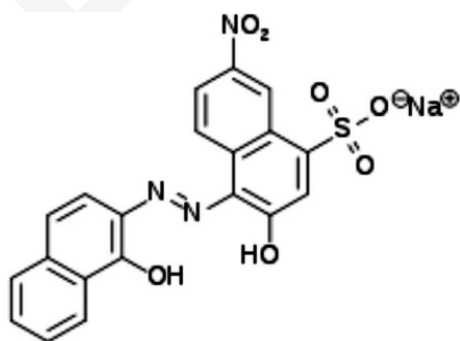
Buffer solutions are considered to possess reserve acidity as well as alkalinity. According to Henderson-Hasselbalch equation, $\text{pH} = \text{pK}_b + \log_{10} [\text{salt}] / [\text{base}]$, At half equivalence point [Concentration of salt = Concentration of base], Therefore $\text{pH} = \text{pK}_b$.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

18. What is buffer solution?

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

19. What is the chemical name of EBT ?

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



20. 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.

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

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



Determination of CaO in the given sample of Cement by Rapid EDTA method

1. What is cement?

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

2. Mention the composition of Portland cement.

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

3. How is cement solution prepared?

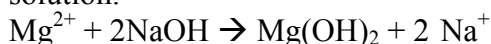
Accurately weighed amount of cement is warmed with moderately conc. HCl till cement dissolves insoluble silica is filtered off & filtrate is the cement solution

4. Why is the titration called rapid EDTA method?

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

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

NaOH reacts with magnesium ion & precipitates as magnesium hydroxide from the cement solution.



6. What are the constituents of cement?

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

7. What is the main constituent of cement?

Calcium oxide.

8. What is the role of glycerol?

Glycerol is added to get sharp end point.

9. What is the role of NaOH?

NaOH is added to mask the magnesium ions.

10. What is the function of diethyleamine?

Diethyleamine is added to maintain a pH of about 12.5.

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

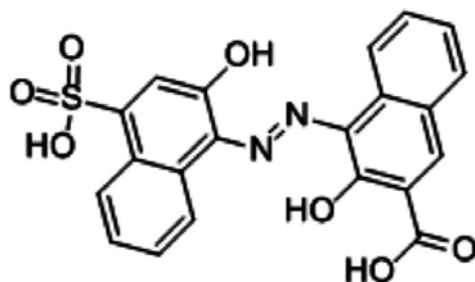
Because it forms a weak complex with calcium ions at pH range of 12-14.

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

Patton and reeder's reagent (Calconcarboxylic acid;

IUPAC name:

Naphthalenecarboxylic acid, 3-hydroxy-4-[(2-hydroxy-4-sulfo-1-naphthalenyl)azo]-; 3-hydroxy-4-[(2-hydroxy-4-sulfo-1-naphthalenyl)azo]-2-Naphthalenecarboxylic acid)



Determination of Percentage of Copper in Brass using standard Sodium thiosulphate solution

1. What is Brass?

Brass is an alloy

2. What is an alloy?

An alloy is a homogeneous mixture of two or more metals

3. What are the constituents of brass?

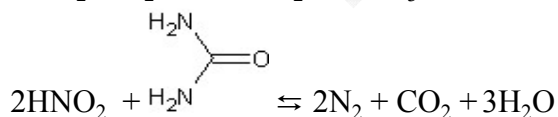
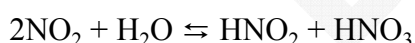
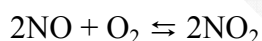
Brass is an alloy of copper (50-90%), zinc (20-40%), small quantities of tin, lead and iron.

4. How is brass solution prepared?

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

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

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



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

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

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

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

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

Cupric hydroxide



9. Why acetic acid is added?

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

10. Why is acidic medium necessary?

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

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

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

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

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

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

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

14. How is liberated iodine estimated?

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

15. What is the reaction that occurs between iodine & sodium thiosulphate?



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

Determination of Iron in the given sample of Haematite ore solution using Potassium dichromate Crystals by external indicator method

1. What is an ore?

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

2. What is a mineral?

A mineral is a naturally occurring metallic compound.

3. What are the main constituents of haematite ore?

Ferric oxide (Fe₂O₃)



4. Give the other forms of iron ore?

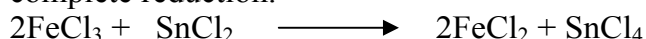
Magnetite (Fe_3O_4), Siderite (FeCO_3), Iron pyrite (FeS_2)

5. Why HCl is added?

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

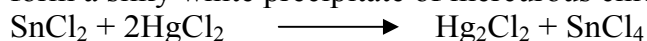
6. What is the role of stannous chloride?

Stannous chloride reduces Ferric ion to ferrous ion and a slight excess is added to ensure complete reduction.



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?

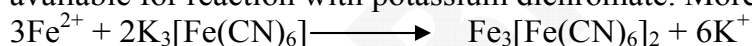
Stannous chloride is a reducing agent. The ferric ions formed during the course of the titration get reduced to ferrous ions. As a result, the volume of potassium dichromate consumed will be more.

9. Which is the indicator used?

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

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

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



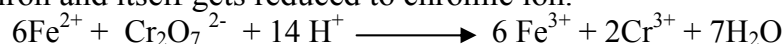
11. Why potassium Ferro cyanide cannot be used as an indicator in the estimation of Fe
Potassium Ferro cyanide cannot be used as an indicator because Ferro cyanide does not react with ferrous ion.

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

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

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

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



14. Can Fe be determined using internal indicators?

Yes, Fe can be determined using internal indicators.

15. Name the internal indicator used for iron determination?

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



Determination of COD of the given Industrial Waste water sample

1. What is sewage?

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

2. What is meant by industrial sewage?

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

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

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

4. What is BOD?

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

5. Why COD is greater than BOD value?

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

6. What are end products of oxidation?

CO₂ & H₂O.

7. Why H₂SO₄ is added to conical flask?

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

8. What is the unit of COD?

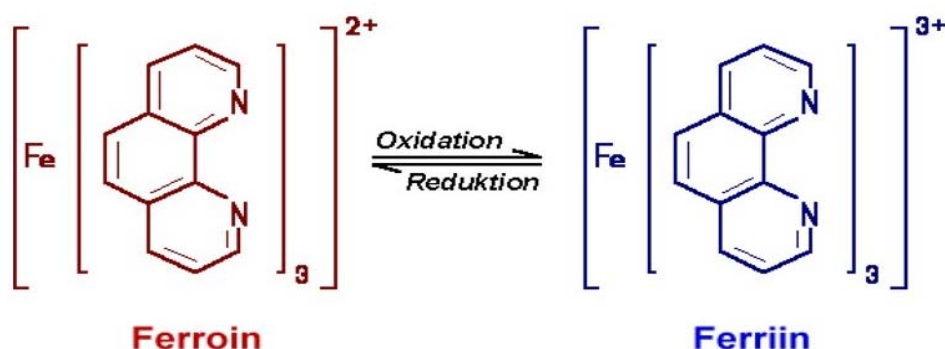
mg/litr of O₂ or mg/dm³ of O₂ or ppm of O₂ .

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

Potassium di chromate.

10. Explain the colour change during the titration.

Ferriin indicator is red in colour in the reduced form with the composition, [Fe(O-Phen)₃]²⁺(ferroin), When ferroin 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 Ferroin which is in red colour which is the end point of titration.



11. 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.

12. 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.

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

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

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

Aromatic hydrocarbons and pyridine are not oxidized in COD test.

15. What is the role of silver sulphate?

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

16. What is the role of mercuric sulphate?

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

17. What are the products formed after COD analysis?

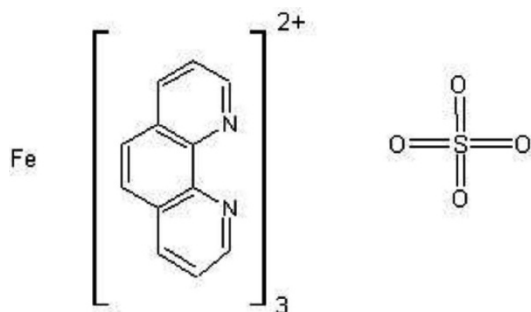
During COD analysis organic matter is completely oxidized to carbon dioxide and water by acidified potassium dichromate solution.

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

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

20. What is the composition of ferroin?

Ferroin is ferrous 1, 10-Phenanthroline sulphate.



Ferrioxalate complex

21. Why blank titration is necessary?

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

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

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

23. What is the limitation of COD?

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



BRIEF PROCEDURE WRITE-UP

PART- A

1. Determination of the viscosity coefficient of a given liquid

Take a clean and dry viscometer and fix the viscometer vertically to a stand. Using a burette transfer a known volume (say 10 ml of the lower bulb) of water through wider limb. Suck the water above the upper mark of the viscometer. Allow it to flow freely through the capillary, when the level of the water just crosses upper mark, start the stop clock and when the water just crosses the lower mark, stop the stop clock. Note down the time of flow in seconds (t_w). The viscometer is washed and rinsed with acetone and dried in oven. The time of flow of given liquid (t_l) between the two markings A and B in seconds is also noted.

The viscosity of the given liquid is calculated using the formula

$$\frac{\eta_l}{\eta_w} = \frac{d_l t_l}{d_w t_w} \quad \text{(Derived from poiseuille's Equation)}$$

$$\eta_l = \frac{d_l t_l}{d_w t_w} \eta_w \quad \eta = \frac{\pi P r^4 t}{8 \nu l} = \frac{\pi h d g r^4 t}{8 \nu l}$$

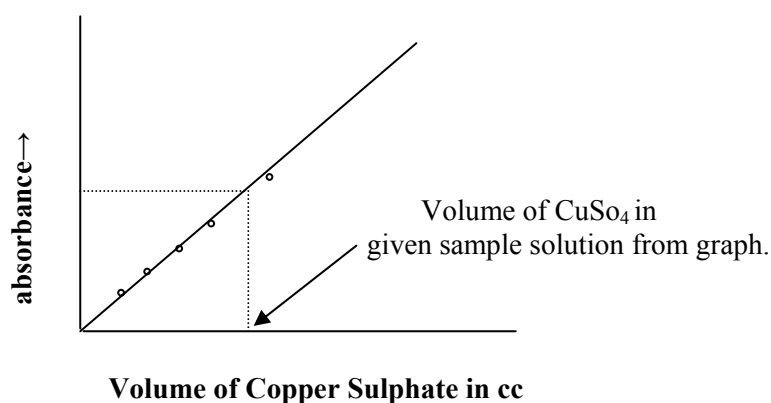
where

d_w, t_w and η_w are the density, time and viscosity of water and
 d_l, t_l and η_l are the density, time and viscosity of the given liquid.

2. Colourimetric estimation of copper

Different standards solutions of copper are prepared as follows:

Exactly 5,10,15,20 cc. and unknown copper sulphate solution are taken in 50 cc. std. flasks. 5 cc of Ammonia solution is added to each of them and made up to the mark. A blank solution is prepared in a separate standard flask by using 5 cc of Ammonia and Water. 620nm Wave length filter is used for recording the optical density of the copper solution. A blank solution is used to set transmission of 100 (O.D =0) in colorimeter. The optical density of the different copper solution is noted. From the linear plot of O.D against the volume of copper solutions the volume of unknown solution is determined.



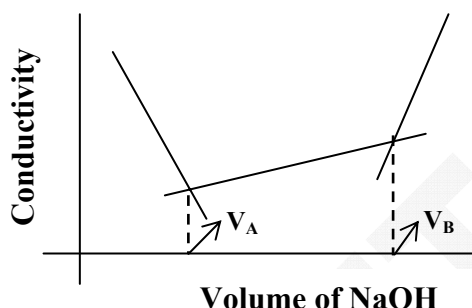
3. Conductometric estimation of HCl in the given acid using standard NaOH solution

Microburette : 1N NaOH

Accessories : conductivity cell connected to a digital conductivity meter.

Beaker : Pipetted out 25cc of the given acid mixture and dip the conductivity cell.

Pipette out 25 ml of acid mixture in to a beaker. Immerse the conductivity cell in to it & connect it to conductivity meter and measure the conductance. Add NaOH from the burette in the increments of 0.5 ml and measure the conductance after each addition. Plot a graph of conductance against volume of NaOH & determine the equivalence point. Calculate the normality & weight of HCl & CH₃COOH in the given so.



where, V_A = Volume of NaOH required to neutralize HCl. $(V_B - V_A)$ = Volume of NaOH required to neutralize acetic acid.

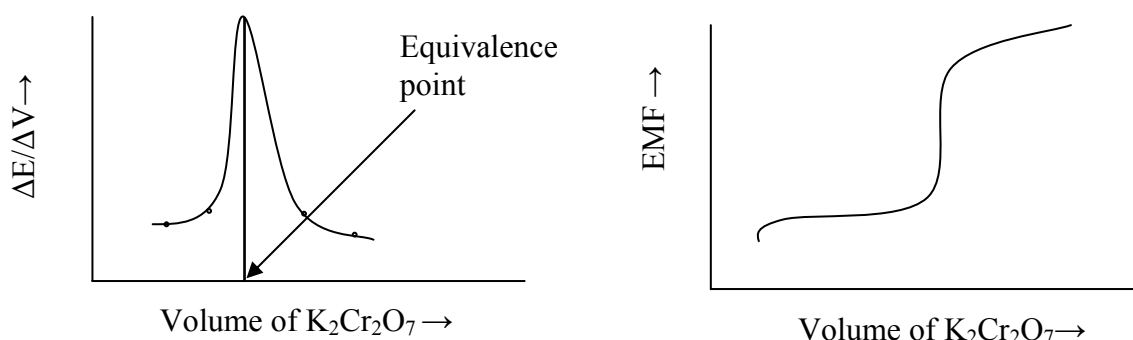
4. Potentiometric estimation of FAS using standard K₂Cr₂O₇ solution

Microburette : Standard K₂Cr₂O₇ solution

Accessories : Calomel + platinum electrode connected to a potentiometer.

Beaker : Pipetted out 25cc of the given FAS solution + 2tt of dil H₂SO₄

FAS solution is pipetted out into a beaker. 2tt of dil H₂SO₄ is added to it. A calomel + platinum electrode is immersed into it and connected the assembly to a potentiometer. FAS is titrated against K₂Cr₂O₇. The potential E is measured for every addition of K₂Cr₂O₇ (0.5 ml) till there is an abrupt increase in the E Value. Addition of K₂Cr₂O₇ (0.5 cc) is continued for 4 more readings. A graph of $\Delta E / \Delta V$ against volume of K₂Cr₂O₇ is plotted. The maxima in graph gives the volume of K₂Cr₂O₇ for equivalence point. From the normality and volume of K₂Cr₂O₇ the weight of FAS in the given solution is calculated.





5. Determination of p^{K_a} value of a weak acid using p^H meter

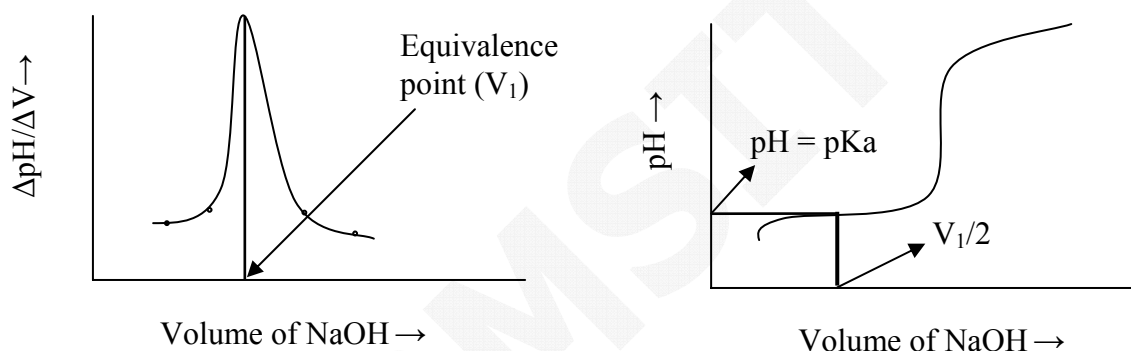
Microburette : 1N NaOH

Accessories : Digital pH meter attached with a combined glass electrode

Beaker : Pipetted out 25cc of the given weak acid and dipped the combined glass electrode into it.

Calibration : Temp setting and pH setting with buffer(pH =7) adjusted.

The weak acid solution is titrated with 1N NaOH solution. The p^H of the solution is recorded after the addition of each 0.25cc till there is an abrupt increase in p^H . Addition of NaOH (0.25 cc) is continued and p^H is noted for 4 more readings. Two graphs of p^H vs volume of NaOH and $\Delta p^H / \Delta V$ vs volume of NaOH are plotted. The maxima in graph two gives the volume of base for equivalence point. Half of this gives the volume of NaOH for half equivalence point. Using this value in graph, one the p^H for this volume is noted. This p^H value is = p^{K_a} of the given weak acid.



PART- B

1. Determination of total hardness of a sample of water.

Part A: Preparation of standard solution of disodium salt of EDTA.

A standard solution of EDTA is prepared by dissolving a known amount of the disodium salt of EDTA with 5cc of ammonia and made up with deionised water in a 250cc standard flask.

$$\text{Molarity of EDTA} = \frac{\text{Weight of EDTA salt} \times 4}{\text{Molecular weight of EDTA salt (372.24)}}$$

Part B: Determination of total hardness of water sample

Burette : Standard EDTA solution

Conical flask : 25 cc water sample + 5 cc of $\text{NH}_3 - \text{NH}_4\text{Cl}$ Buffer

Indicator : Eriochrome Black T

End point : Wine red to clear blue

Calculations : From the volume of EDTA consumed, the **Total hardness of the given water sample (expressed as CaCO_3)** is calculated.



2. Determination of calcium oxide in the given sample of cement solution

Part A: Preparation of standard solution of disodium salt of EDTA.

A standard solution of EDTA is prepared by dissolving a known amount of the disodium salt of EDTA with 5cc of ammonia and made up with deionised water in a 250cc standard flask.

$$\text{Molarity of EDTA} = \frac{\text{Weight of EDTA salt} \times 4}{\text{Molecular weight of EDTA salt (372.24)}}$$

Part B: Estimation of CaO in cement

Burette	: Standard EDTA solution
Conical flasks	: 25 cc of cement solution + 10cc of 4N NaOH+5cc glycerine +5cc of diethyl amine
Indicator	: Patton and Reeder's indicator
End point	: Wine red to clear blue

Calculations : From the volume of EDTA consumed, the Percentage of CaO in the given cement solution is calculated.

3. Determination of percentage of copper in brass using standard sodium thiosulphate solution

Part A: Preparation of the brass solution

A known weight of the brass sample is dissolved in minimum amount of nitric acid. 1g of urea is added and boiled till all the oxides of nitrogen escape. The solution is cooled and ammonium hydroxide is added dropwise till a bluish white precipitate appears. Added acetic acid till the original blue colour appears and made upto 250cc with deionised water.

Part B: Estimation of Copper.

Burette	: Standard sodium thiosulphate solution
Conical flasks	: 25 cc of brass solution + 1 test tube of 10 % KI + 1 test tube water.
Indicator	: 2 or 3 drops starch solution near the end point (when the solution acquires pale yellow colour)
End point	: Disappearance of the blue colour to milky white colour.

Calculations : From the volume of thio consumed, the **Percentage of Copper** in the given brass sample is calculated.

4. Determination of iron in a given sample of Haematite Ore Solution

Part A: Preparation of standard solution of potassium dichromate.

A standard solution of $\text{K}_2\text{Cr}_2\text{O}_7$ is prepared by dissolving 1.2 g of $\text{K}_2\text{Cr}_2\text{O}_7$ and made up with deionised water in a 250cc standard flask.



$$\text{Normality of } K_2Cr_2O_7 = \frac{\text{weight of } K_2Cr_2O_7 \times 4}{\text{Eq. wt. of } K_2Cr_2O_7} = \frac{\text{ } \times 4}{49}$$

Part B: Estimation of Iron.

Burette : Standard $K_2Cr_2O_7$ solution

Conical flasks : 25 c.c of iron ore solution + $\frac{1}{4}$ t.t of con.HCl. Heat and add $SnCl_2$ dropwise till colourless . Cool and add $HgCl_2$ till white precipitate appears(grey ppt or no ppt reject the solution).

Indicator : $K_3Fe(CN)_6$ as external indicator

End point : No change in the colour of indicator when a drop of reaction mixture is brought in contact with it.

Calculations : From the volume of $K_2Cr_2O_7$ consumed, the **Percentage of iron** in the given haematite sample is calculated.

5. Determination of Chemical oxygen demand (COD) of the waste water

Part A: Preparation of standard solution of ferrous ammonium sulphate(Mohrs Salt)

A standard solution of FAS is prepared by dissolving a known weight of FAS with 10cc of 1:1 sulphuric acid and made up with deionised water in a 250cc standard flask.

$$\text{Normality of FAS} = \frac{\text{weight of FAS} \times 4}{\text{Eq. wt. of FAS}} = \frac{\text{ } \times 4}{392} = Z$$

Part B : Back Titration

Burette : Standard FAS solution

Conical flask : 25 cc of waste water + 25 cc $K_2Cr_2O_7$ solution + $\frac{3}{2}$ t.t of H_2SO_4

Indicator : **2 drops of ferroin**

End point : Appearance of brown colour

Volume of FAS used for back titration =(V_2) cc
(i.e., the volume of FAS consumed by unreacted $K_2Cr_2O_7$ solution)

A blank titration is performed in the same way as above without the waste water.

From the difference in the titre value the COD of the waste water sample is calculated



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PRACTICAL EXAMINATION

SCHEME OF VALUATION

Subject : Engg. Chemistry

Lab. Subject code : 18CHEL16/26

Description	Max. Marks	Part A marks	Part B Marks
Procedure write up	15	08	07
Conduction	52	26	26
Calculation, Graph works and result	18	09	09
Viva-Voce	15	07	08

PART – A : INSTRUMENTAL					
Potentiometry, Colorimetry & Flame Photometry(Na/K)		pK _a and Viscosity		Conductometry	
Error (cm ³)	Marks	Error (%)	Marks	Error (cm ³)	Marks
± 0.5	26	± 5.0	26	± 0.5	13 + 13
± 0.51 to 0.6	22	± 5.1 to 6.0	22	± 0.51 to 0.6	11+11
± 0.61 to 0.7	18	± 6.1 to 7.0	18	± 0.61 to 0.7	9+9
± 0.71 to 0.8	14	± 7.1 to 8.0	14	± 0.71 to 0.8	7+7
± 0.81 to 1.0	10	± 8.1 to 10.0	10	± 0.81 to 1.0	5+5
> ± 1.0	Zero	> ± 10.0	Zero	> ± 1.0	Zero
Graph : 5 marks Calculation : 4 marks		pK _a : Two Graphs : 5 + 4 Marks Viscosity : Calculation : 9 marks		Graph : 5 marks Calculation : 4 marks	

PART – B : VOLUMETRY	
Total Hardness, CaO in Cement, Cu in Brass, Fe in Haematite, COD, Total alkalinity,	
Error (cm ³)	Marks
± 0.2	13 + 13
± 0.3	11 + 11
± 0.4	9 + 9
± 0.5	7 + 7
± 0.6	5 + 5
> ± 0.6	Zero
Calculation : 9 Marks	
Note : Best Two(out of Three) TITRE values should be considered for valuation	



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