|| JAI SRI GURUDEV ||

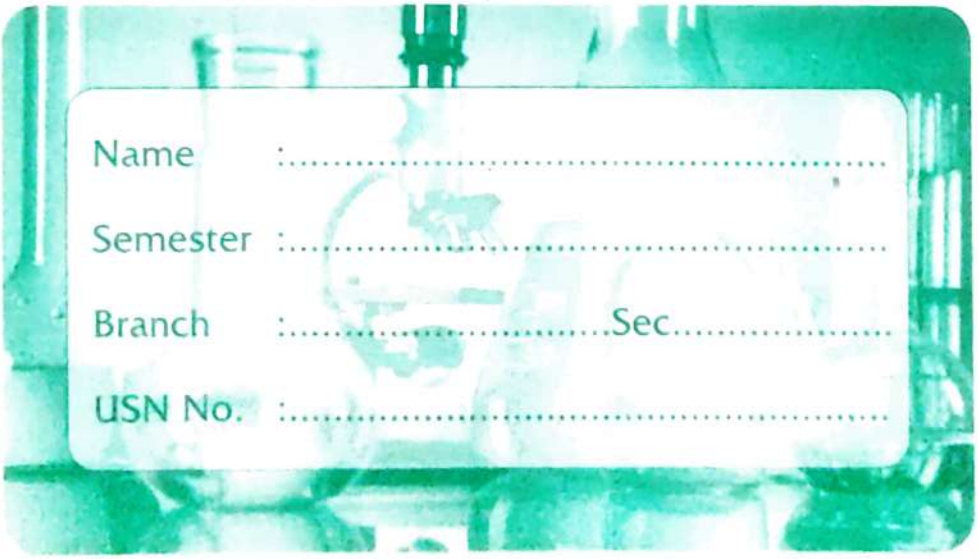
SRI ADICHUNCHANAGIRI SHIKSHANA TRUST (R)

SRI JAGADGURU CHANDRASHEKARANATHA SWAMIJI INSTITUTE OF TECHNOLOGY

CHICKBALLAPUR - 562 101.

CHEMISTRY LABORATORY MANUAL

(As per VTU Syllabus)



Name :...

Semester :...

Branch :............................Sec.........................

USN No. :...

DEPARTMENT OF CHEMISTRY

SRI JAGADGURU CHANDRASHEKARANATHA SWAMIJI INSTITUTE OF TECHNOLOGY

Affiliated to VTU, Belagavi, Approved by AICTE, New Delhi,

Accredited by NAAC and ISO-9001:2015 Certified

P.B. No. 20, B. B. Road, Chickballapur - 562101

Phone : 08156 - 263181 / 82 / 83 Web : www.sjcit.ac.in.

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Course Objectives:

To provide students with practical knowledge of

- Quantitative analysis of materials by classical methods of analysis.
- Instrumental methods for developing experimental skills in building technical competence.

Course Outcomes:

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

- Handling different types of instruments for analysis of materials using small quantities of materials involved for quick and accurate results, and
- Carrying out different types of titrations for estimation of concerned in materials using comparatively more quantities of materials involved for good results.

VISION AND MISSION OF THE COLLEGE

VISION

- Preparing Competent Engineering and Management Professionals to serve the society.

MISSION

- Providing students with a sound knowledge in fundamentals of their branch of study.
- Promoting excellence in Teaching, Training, Research and Consultancy.
- Exposing students to Emerging Frontiers in various domains enabling Continuous Learning.
- Developing Entrepreneurial acumen to venture into innovative areas.
- Imparting value based professional education with a sense of Social Responsibility.

VISION AND MISSION OF THE DEPARTMENT

VISION

- Amalgamating chemistry concepts with the trending technologies in engineering disciplines.

MISSION

- Training the students to enrich knowledge in energy storage systems, corrosion control techniques, metal finishing, nano technology & renewable source of energy.
- Building advanced knowledge in nano technology among students to focus on research.
- Encouraging students to solve societal problems with innovative thought process.

Course Objectives:

To provide students with practical knowledge of

- Quantitative analysis of materials by classical methods of analysis.
- Instrumental methods for developing experimental skills in building technical competence.

Course Outcomes:

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

- Determine the pKa and coefficient of Viscosity of a given organic liquid.
- Estimate the amount of substance present in the given solution using Potentiometer Conductometric and Colorimetric analysis
- Determine the total hardness and chemical oxygen demand in the given water sample by volumetric analysis method
- Determine the percentage of Cao, copper, and Iron in the given analyte solution by titration method.
- Demonstrate flame photometric estimation of sodium & potassium and the synthesis of nanomaterials by Precipitation method.

CHEMISTRY PRACTICALS

Expt. No.	PARTICULARS	Page No	Date	Signature	
	Instrumental experiments			Student	Staff
1	Potentiometric estimation of FAS using standard $K_2Cr_2O_7$ solution.				
2	Conduct metric estimation of the acid mixture.				
3	Determination of Viscosity coefficient of a given liquid using Ostwald's viscometer				
4	Colorimetric estimation of copper.				
5	Determination of pKa value of a given weak acid using a pH meter				
	Volumetric experiments				
1	Estimation of Total hardness of water by EDTA complexometric method.				
2	Estimation of CaO in cement solution by rapid EDTA method.				
3	Determination of percentage of copper in brass using standard sodium thiosulphate solution.				
4	Determination of Chemical oxygen demand of industrial wastewater.				
5	Estimation of percentage of iron in the given rust solution using standard Potassium Dichromate solution (External indicator method)				
	Demonstration Experiments (For CIE)				
1	Flame photometric estimation of Sodium & Potassium.				
2	Synthesis of nanomaterial by Precipitation method.				
	MODEL PROCEDURES				
	VIVA -VOCE QUESTIONS AND ANSWERS				

Instrumental Experiments

EXPERIMENT NO: 1

Potentiometric estimation of FAS using standard $K_2Cr_2O_7$ solution.

Aim : To estimate the amount of FAS in a given solution.

Theory: Redox titrations can be carried out potentiometrically using platinum-calomel electrode combination in a manner similar to acid – base titration. For the reaction.

$$\text{Reduced form} \rightarrow \text{oxidized form} + n \text{ electrons}$$

The potential is given by Nernst equation,

$$E = E^0 + \frac{0.0591}{n} \log \frac{[\text{oxidized form}]}{[\text{reduced form}]}$$

Where E^0 is the standard electrode potential of the system.

A Potentiometric titration may be defined as a titration in which the end point is detected by measuring the change in potential of an electrode which responds to the change in concentration of ions during the titration. The electrode which responds to the change in concentration of the ion in the solution is called the indicator electrode (Platinum electrode), the indicator electrode is combined with a reference electrode (Calomel electrode) to form a cell and emf of the so formed cell is measured during the titration. The emf of the cell changes gradually during titration till the equivalence point and changes rapidly at the equivalence point and again the change is gradually after the equivalence point. When emf, $\Delta E / \Delta V$ is plotted against volume of $K_2Cr_2O_7$ (potassium dichromate) solution is shown in the figure .The sudden change in the potential at the equivalence point is explained as follow,

The reaction that takes place in the determination of Fe^{2+} is

$$(Fe^{2+} \rightarrow Fe^{3+} + e^-) \text{ x6}$$

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$$

$$6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \rightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$$

Prior to the equivalence point the potential is determined by the Fe^{3+}/Fe^{2+} system and the potential is given by the equation,

$$E_{cell} = E^0_{Fe} = E^0_{Fe^{3+}/Fe^{2+}} + \frac{0.059}{1} \log \frac{[Fe^{3+}]}{[Fe^{2+}]}$$

$$= 0.75V + 0.0591 \log \frac{[Fe^{3+}]}{[Fe^{2+}]}$$

The potential of the solution will be around 0.75V (since the contribution to the potential by the second term is negligible). At the equivalence point the potential is determined by both $E^0_{Fe^{2+}}$ and $E^0_{Cr2O7^{2-}}$ and is given by

$$E_{cell} = \frac{E^0_{Fe^{2+}} + E^0_{Cr2O7^{2-}}}{2} = \frac{0.75V + 1.33\ V}{2} = 1.04\ V$$

Observation & Calculations:

Volume of $K_2Cr_2O_7$ added in cm^3	E (mv)	ΔE	ΔV	$\frac{\Delta E}{\Delta V}$
0.0		…….	…….	…….
0.5			0.5	
1.0				
1.5				
2.0				
2.5				
3.0				
3.5				
4.0				
4.5				
5.0				
5.5				
6.0				
6.5				
7.0				
7.5				
8.0				

Calculations:

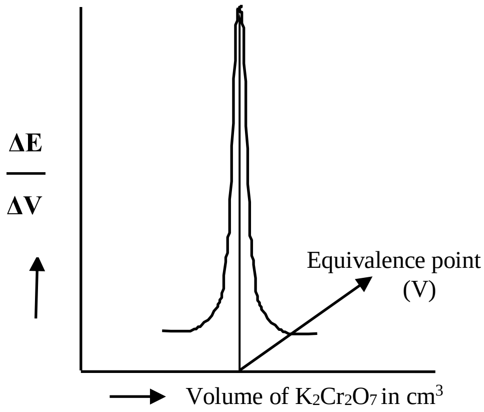
Volume of FAS given , $V_1 = 25cm^3$

Normality of $K_2Cr_2O_7$, $N_2 = 0.5$ N

$(N_1V_1)_{FAS} = (N_2V_2)_{K2Cr2O7}$

$N_1 = N_2V_2 / V_1$

=…………………. =………………..N



Normality of FAS, N_1 =………………..N

Wt of FAS/liter = $N_{(FAS)}$ x Eq.wt of FAS(392)

= ……………………………………………=…………………………………g/lit.

Therefore, Weight of FAS/liter =……………g/lit

Beyond the equivalence point the potential is determined by $Cr_2O_7^{2-}/Cr^{3+}$ system given by the equation,

$$E_{cell} = E_{Cr} = E^0_{Cr2O7^{2-}/Cr3+} + 0.0591 \log \left[\frac{Cr_2O_7^{2-}}{Cr^{3+}} \right]$$

$$E_{cell} = 1.33V + \frac{0.0591}{6} \log \left[\frac{Cr_2O_7^{2-}}{Cr^{3+}} \right]$$

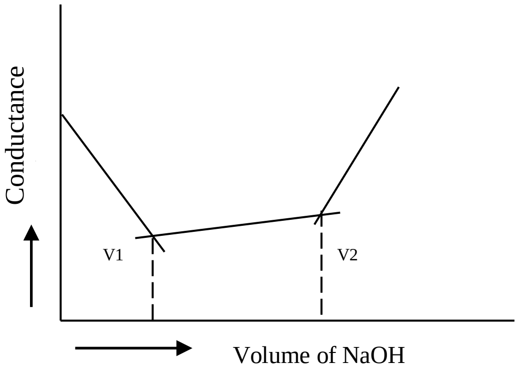
Thus an abrupt increase in the potential of the solution in the vicinity of the equivalence point is observed. This marks the equivalence point. In the experiment, the potential of the cell is determined with reference to saturated calomel electrode.

Procedure: Transfer $25cm^3$ of ferrous ammonium sulphate solution into a beaker using the aid of a pipette. Add two test tube full of dilute sulphuric acid. Immerse the electrode assembly (platinum & calomel) into the solution, connect the electrodes to a potentiometer and measure the potential. Fill the burette with potassium dichromate solution. Add $0.5\ cm^3$ of $K_2Cr_2O_7$ into the beaker. Stir the solution carefully and measure the potential. Continue the procedure till potential shows a tendency to increase rapidly and note down few readings after rapid increase. Determine the end point by differential method. i.e.,by plotting $\Delta E / \Delta V$ against volume of $K_2Cr_2O_7$ as shown in the figure. Calculate the normality of the FAS solution and determine the amount of FAS present in the given solution.

RESULT: **Amount of FAS present in a given solution is = ……………grams/liter.**

Observation and calculation:

Volume of NaOH added in cm^3	Conductance Ώ$^{-1}$cm^{-1}
0.0	
0.5	
1.0	
1.5	
2.0	
2.5	
3.0	
3.5	
4.0	
4.5	
5.0	
5.5	
6.0	
6.5	
7.0	
7.5	
8.0	
8.5	
9.0	
9.5	
10.0	
10.5	
11.0	
11.5	
12.0	



EXPERIMENT NO: 2

Conductometric estimation of the acid mixture

Aim:- Conductometric estimation of Acid mixture

Theory: Measurement of conductance can be employed to determine the end point in acid base titration. In Conductometric titration, there is a sudden change in conductance of the solution at the equivalence points. The principle underlying Conductometric titration is the substitution of ions one mobility by ions of another mobility. Therefore, the conductance of solution depends on the no mobility of ions. The equivalence point is determined graphically by plotting conductance against volume of NaOH.

Conductometric titration may be applied for the determination of acids present in a mixture. In the titration of the mixture of a weak acid (CH_3COOH) and a strong acid (HCl) with a strong base (NaOH). The conductance decreases upon adding NaOH to acid mixture owing to the substitution of higher mobile H^+ ions (mobility: 350 $ohm^{-1}m^{-1}$) by less mobile Na^+ion (mobility: 50 $ohm^{-1}m^{-1}$). This trend continues till all the H^+ ions of HCl replaced ie. the strong acid is completely neutralized. Continuation of addition of NaOH raises the conductance moderately, as the weak acid CH_3COOH is converted into its salt CH_3COONa. Further addition of NaOH raises the conductance steeply due to the presence of free OH^- ions (mobility: 198 $ohm^{-1}m^{-1}$). The titration curve depicting the location of the equivalence points is shown in figure (graph).

$$[H^+ + Cl^-] + [Na^+ + OH^-] \longrightarrow [Na^+ + Cl^-] + H_2O$$

$$[CH_3COO^- + H^+] + [Na^+ + OH^-] \longrightarrow [CH_3COO^- + Na^+] + H_2O$$

Procedure: Pipette out 50cm^3 of acid mixture into a100cm^3 beaker. Immerse the conductivity cell in acid mixture and measure the conductance without adding NaOH solution . Now add sodium hydroxide (NaOH) from a burette in the increments of 0.5$cm^{3,}$ stir the solution gently and measure the conductance after each addition. Plot a graph of conductance against volume of NaOH on the X axis to get three straight lines. The point of intersection of the first & second lines gives the volume of NaOH needed to neutralize only HCl (V_1). The point of intersection of the second& third lines gives the volume of NaOH needed to neutralize only CH_3COOH (V_2).

Result:- Amount of HCl present in a given acid mixture =…………………g/lit

Amount of CH_3COOH present in a given acid mixture =……………g/lit

Estimation of HCl:

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

$$\text{Normality of HCl} = \frac{\text{Normality X Volume of NaOH}}{50} = \text{--------------------}$$

= ---------------------N

Therefore the weight of HCl/lit = Normality of HCl x Eq.wt.ofHCl(36.5)

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

= -----------------------gms

Estimation of CH_3COOH:

Volume of NaOH required to neutralize CH_3COOH = (V_2-V_1) = ------------------

= -----------cm^3

$$\text{Normality of } CH_3COOH = \frac{\text{Normality x Volume of NaOH}}{50} = \text{-------------------}$$

= -----------------N

Therefore the weight of CH_3COOH /lit = Normality of CH_3COOH x Eq .wt .of CH_3COOH (60)

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

=-----------------------gms

Observation & Calculations:

	Time of flow (seconds)			
	Trial 1	**Trial 2**	**Trial 3**	**Average**
Water				
Test liquid				

Calculations:

where,

$\frac{\eta_1}{\eta_2} = \frac{t_1 d_1}{t_2 d_2}$

η_1 = viscosity co-efficient of water = ……… milli poise

d_1=density of water = ……………….g/cm^3

$\eta_2 = \frac{t_2 d_2}{t_1 d_1} \times \eta_1$

d_2=density of test liquid = ……………g/cm^3

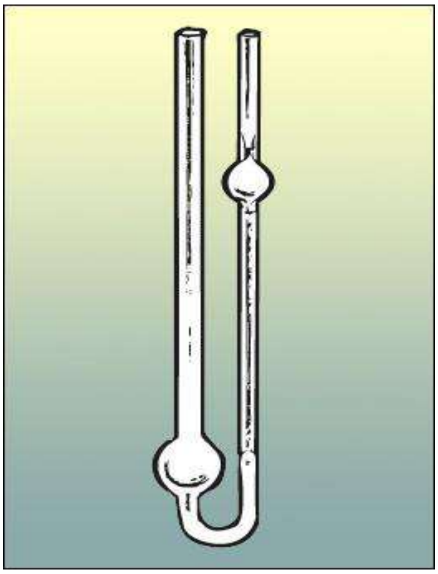
t_1 = time flow of water =…………..seconds.

t_2 = time flow of test liquid.………..seconds.

= ………………….................-.............................

= ………………………………..…………

η_2 = …………………….... milli poise



EXPERIMENT NO: 3

Determination of viscosity co-efficient of a given liquid using Ostwald's viscometer.

Aim: To determine the Viscosity co-efficient of a given liquid.

Theory: Viscosity arises due to internal friction between moving layers of molecules. A liquid flowing through a cylindrical tube of uniform diameter is expected to move in the form of molecular layers. A layer close to the surface is almost stationary while that at the axis of the tube moves faster than any other intermediate layer. A slow moving layer exerts a friction on its nearest moving layer. This property of a liquid by which it opposes motion between layers is called Viscosity. **The co-efficient of viscosity** is defined as the tangential force per unit area required to maintain a unit velocity gradient between any two successive layers of a liquid situated unit distance apart. The co-efficient of viscosity of a liquid is given by Poiseuille's formula,

$$\eta = \pi P r^4 t / 8vl$$

Where V = volume of the liquid, r = radius of the tube, l = length of the tube, ρ is the pressure difference between the two ends of the tube, η is the co-efficient of viscosity of the liquid and t is the time of flow. If equal volumes of two different liquids are allowed to flow through the same capillary under identical conditions, then,

$$\eta_1 / \eta_2 = t_1 d_1 / t_2 d_2$$

The time (t_2) taken by the test liquid to flow through a certain distance in the capillary is determined. The time (t_1) taken by a standard liquid to flow through the same distance is measured. Co-efficient of viscosity of the test liquid is calculated from the densities (d_1 and d_2) of the test liquid and the standard liquid and the co-efficient of viscosity (η_1) of the standard liquid. The viscosity measurement is carried out in Ostwald's type capillary viscometer shown in figure. Viscosity is a temperature dependent property and hence the measurements are carried out at constant temperature.

Procedure: Clean the viscometer with water, rinse with acetone and dry it. Fix the viscometer vertically to stand in a constant temperature bath (beaker containing water). Using a clean pipette, transfer a known volume (say 10 cm^3) of water into the wider limb. Allow the viscometer to stand for some time so that the liquid attains the temperature of the bath. Suck the water above the upper mark of the viscometer and allow it to flow freely through the capillary. When the level of water just crosses upper mark, start a stop clock. Stop the clock when the water just crosses the lower mark. Note the time of flow in seconds (t_1). Repeat the experiment to get agreeing values. Remove the water from the viscometer and rinse the viscometer with acetone and dry it. Repeat the experiment with exactly the same volume of the test liquid whose viscosity co-efficient is to be determined and find the time to flow (t_2 seconds) of the test liquid. Calculate the co-efficient of viscosity of the liquid.

Result: **Viscosity co-efficient of a given test liquid is =…………………milli poise.**

EXPERIMENT NO: 4

Colorimetric estimation of Copper

Aim: Estimation of copper in a given copper sulphate solution calorimetrically.

Theory: When a monochromatic light of intensity I_0 is incident on a transparent medium, a part I_a is absorbed, a part I_r is reflected and the remaining part 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, $\log 1/T = \log I_0/ I_t$ is called the absorbance or optical density A. The relation between absobance A, concentration c (expressed in mol lit^{-1}) and path length t (expressed in cm) is given by Beer – Lambert's law (Variation of absorbance is directly proportional to concentration),

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

Where ε is the molar extinction co-efficient, ε is a constant for a given substance at a given wavelength. If the path length (cell thickness) is kept constant, then, A α c, hence a plot of absorbance against concentration gives a straight line as shown in figure (Calibration curve). The colour is measured using a spectrophotometer at a wavelength where the absorbance is maximum.

A series of standard solutions containing cupric ions is treated with ammonia to get deep blue cuprammonium complex and is diluted to definite volume.

$$Cu^{2+} + 4\,[NH_3] \rightarrow \underset{\text{Deep blue}}{[Cu(NH_3)_4]^{2+}}$$

The absorbance of each of these solutions is measured at 620 nm since the complex shows maximum absorbance at this wavelength. The volume is plotted against absorbance to get a calibration curve. A known volume of the test solution ($CuSO_4$) 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 volume is determined from the calibration curve.

Observation & calculations:

Volume of $CuSo_4$ incm^3	**Absorbance**
2.5	
5.0	
7.5	
10.0	
12.5	
Test solution	

Calculations:

Let ' X ' be the volume of test solution, (from the graph) = …………………cm^3

1cm^3 of $CuSo_4$ solution contains 0.004g of $CuSo_4$

Therefore `X' $cm^3$of test solution contains X x 0.004 =………………..

=………….. `Y' g of $CuSo_4$ (Salt)

249 gms (M wt. of $CuSo_4$) of Copper sulphate contains 63.54 gm of Copper

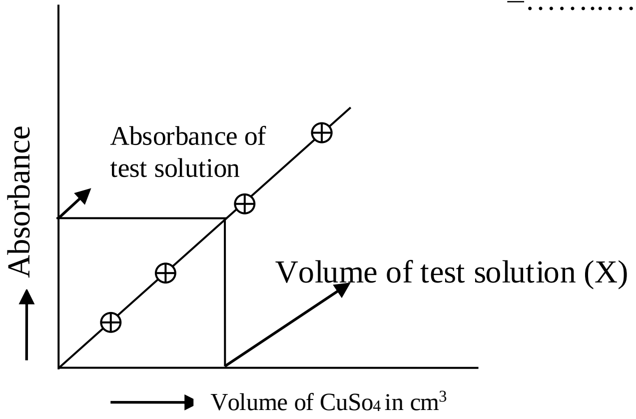
`Y' gms of $CuSo_4$ (Salt) contains = Y x 63.54 / 249

=……………………

=…………..gms (Z)

Therefore, amount of copper present in a given test ($CuSO_4$) solution= Zx1000 mg

=……..………mg



Procedure:

Fill the burette with $CuSO_4$ solution and transfer 2.5, 5.0, 7.5, 10.0 & 12.5 cm^3 $CuSO_4$in labeled 25 cm_3volumetric flasks. Add 2.5 cm^3 of ammonia solution to each of them and make up to the mark with ion exchange water. Stopper the volumetric flasks and mix the solutions well. To the test solution taken in a 25cm^3 volumetric flask, add 2.5 cm^3 of ammonia and make up to the mark with water Mix well. Prepare a blank solution by diluting 2.5cm^3 of ammonia solution in a 25cm^3 volumetric flask to the mark with ion exchange water and mixing well. After 10 minutes, measure the absorbance of the solutions against blank solution at 620 nm using a spectrophotometer. Tabulate the readings as shown. Draw a calibration curve by plotting volume of copper sulphate against absorbance. Using the calibration curve, determine the volume of copper sulphate in a given test solution and calculate the amount of copper present in a given test solution.

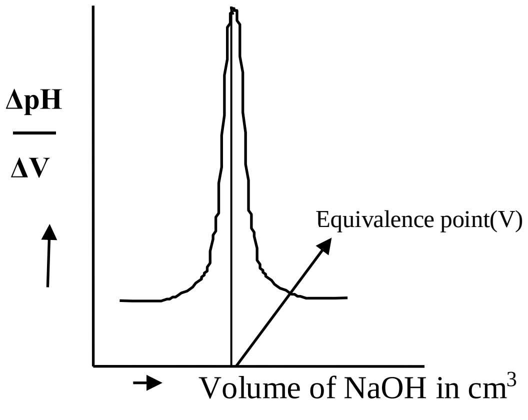
Note: Ammonia should be added to all the solutions including the test solution at the same time.

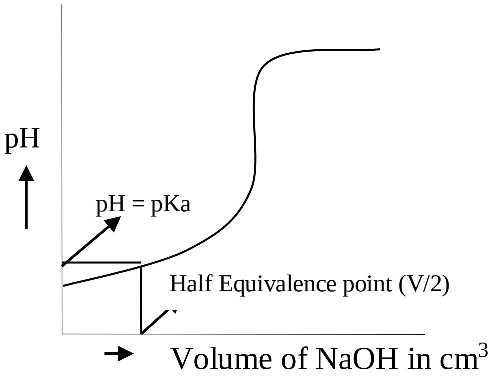
RESULT: **Amount of copper present in a given test ($CuSO_4$) solution is = ………..mg.**

Observation & Calculations:

Tabular Column

Volume of NaOH added in cm^3	**pH**	**ΔpH**	**ΔV**	$\frac{\Delta pH}{\Delta V}$
0.0		…….	…….	…….
0.5			0.5	
1.0				
1.5				
2.0				
2.5				
3.0				
3.5				
4.0				
4.5				
5.0				
5.5				
6.0				
6.5				
7.0				
7.5				
8.0				





EXPERIMENT NO: 5

Determination of pKa of a weak acid using pH meter.

Aim: To determine the pKa value of weak acid.

Theory: Weak acid is a weak electrolyte and undergoes only partial dissociation in the solution, for example, acetic acid when dissolved in water

$$CH_3COOH \rightleftharpoons CH_3COO^- + H^+$$

$$\text{The dissociation constant, } Ka = \frac{[H^+][CH_3COO^-]}{[CH_3COOH]}.$$

$$pKa = -\log_{10}Ka$$

Higher the value of pKa, lower is the K_a value of the acid.

pKa value of a weak acid can be determined by the pH titration method. In this method a known volume of the weak acid is titrated against a strong base, and pH of the solution is measured after each addition of the base. During the titration, the pH of the solution increases gradually till very near to equivalence point and there is a sharp increase in pH at the equivalence point. After the equivalence point the increase in pH is again very gradual. Therefore, the equivalence point is indicated by the sharp increase in pH. The equivalence point is determined by plotting volume of the base against $\Delta p^H/\Delta V$, as indicated by the volume corresponding to the maximum of the curve.

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 pH of a such a mixture is given by Henderson-Hasselbalch equation,

$$pH = pKa + \log\frac{[Salt]}{[Acid]}$$

At half equivalence point, [salt] = [acid] and hence pH = pKa. Thus P^H half equivalence point gives the pKa of weak acid.

Procedure: Transfer 25 $cm^3$of the given weak acid (acetic acid) into a beaker using a pipette. Immerse a glass electrode calomel electrode assembly (combined electrode) into the acid and connect to a pH meter. Measure the pH of the acid. Fill a burette with the base (Sodium hydroxide).Add 0.5cm^3 of the base to the acid. Stir the solution carefully and measure the pH after each addition. Continue the titration till pH shows a tendency to increase rapidly and note down few readings after rapid increase .Tabulate the readings as given below.

Plot a graph of $\Delta pH/\Delta V$ against volume of sodium hydroxide added and determine the equivalence point and plot a graph of pH against the volume of Sodium hydroxide added. Determine the equivalence point and hence the pH at half the equivalence point (V/2). This gives the pKa of the acid.

RESULT: **pKa of given weak acid is = ……………………**

OBSERVATION & CALCULATIONS:

Weight of EDTA + bottle = ……………grams

Weight of empty bottle = ……………..grams

Weight of EDTA = ……………………..grams

Molarity of EDTA = $\dfrac{\text{Weight of EDTA x 4}}{\text{Molecular Weight of EDTA (372)}}$ =……………….

……………….M(Z)

Tabular column

Trial No.	1	2	3
Final burette reading			
Initial burette reading			
Volume of the EDTA rundown in cm^3			

CALCULATIONS:

Volume of EDTA required to react with $25cm^3$ of hard water =………………(X) cm^3

$1000cm^3$ of 1M EDTA= 100 grams of $CaCO_3$. = Molecular mass of $CaCO_3$ =100

Xcm^3 Z M EDTA = $\dfrac{X \times Z \times 100}{1000}$ =………………… = …………………….(a)

Therefore 10^6 cm^3 of hard water sample contains = $\dfrac{a \times 10^6}{25}$ =……….............

=……………ppm of $CaCO_3$

Hardness of the given hard water sample is =………………………. ppm of $CaCO_3$

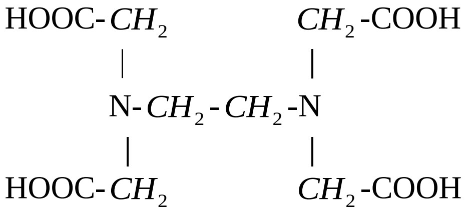
Volumetric experiments

EXPERIMENT NO:1

Estimation of Total hardness of water by EDTA complexometric method.

Aim: To determine the hardness of a given water sample

Theory: Hardness of water is due to the presence of dissolved salts of calcium and magnesium. The hardness is determined using ethylene diamine tetra acetic acid (EDTA) and is expressed in terms of parts per million (ppm) of $CaCO_3$. EDTA is a hexadentate ligand and forms complex with Ca^{2+} and Mg^{2+} ions. The molecule represented as H_4Y has two easily replaceable hydrogen atoms in the molecule. EDTA has the structure.



$$\begin{array}{ccc} \text{HOOC-}CH_2 & & CH_2\text{-COOH} \\ | & & | \\ \text{N-}CH_2 & \text{-} & CH_2\text{-N} \\ | & & | \\ \text{HOOC-}CH_2 & & CH_2\text{-COOH} \end{array}$$

The ionization in solution is represented as

$$H_4Y \rightarrow H_2Y^{2-} + 2H^+$$

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

$$M^{2+} + H_2Y^{2-} \rightarrow MY^{2-} + 2H^+$$

Where M^{2+} is Ca^{2+} or Mg^{2+} .

The hardness of water can be determined by titrating a known volume of water against standard EDTA solution at a pH-10 using Eriochrome black-T as indicator. The colour of free indicator at pH-10 is blue. Eriochrome black-T forms a wine red complex with M^{2+} which is relatively more stable than MY^{2-}.On titrating EDTA first reacts with free M^{2+} ions and gives indicator complex. The latter gives a colour changes from wine red to blue at the equivalence point. Such indicators referred to a Metal-ion indicators. However, this indicator cannot give a sharp colour change in the absence of magnesium ions. Since the reaction involves the release of H^+ ions, a buffer mixture is used to maintain a pH of 10. The buffer mixture used in the titration is NH_3 –NH_4Cl. The free acid (EDTA) is only sparingly soluble, hence the more soluble disodium salt, Na_2H_2Y, is used for preparing the standard EDTA solution.

M^{2+}+EBT → M – EBT Complex
(Wine red)
M – EBT Complex + EDTA → M-EDTA Complex + EBT
(Blue)

Procedure:

Part-A: **Preparation of standard EDTA solution.**

Weigh around 1.5g of disodium salt of EDTA crystals using an electronic balance and transfer the crystals carefully into 250cm^{3}volumetric flask through a funnel and find the weight of the empty bottle. Add 3 cm^3 of 1:1 NH_3 and ion exchange water through the funnel to ensure that all the crystals slide down into the flask. Wash the funnel with ion exchange water and remove the funnel. Dissolve the crystals by swirling the flask Dilute with water and make up to the mark. Stopper and invert the flask several times so that a homogeneous solution results. Calculate the molarity of EDTA.

Part-B: Determination of total hardness of a water sample.

Transfer 25cm^3 of the given water sample into a clean conical flask using a pipette. Add 3 cm^{3}of NH_3–NH_4Cl buffer and indicator 3 to 4 drops of EBT and titrate against EDTA till the wine red colour disappears and blue colour appears. Repeat the experiment for agreeing values.

Result: Total hardness of the given water sample is =…………………… ppm of $CaCO_3$.

OBSERVATION AND CALCULATIONS:

Weight of EDTA + bottle =.. grams.

Weight of empty bottle=... grams.

Weight of EDTA= ………………………..grams

Molarity of EDTA = $\dfrac{\text{Weight of EDTA x 4}}{\text{Molecular Weight of EDTA (372)}}$ =……………….

……………….M(Z)

Tabular column

Trial No.	1	2	3
Final burette reading			
Initial burette reading			
Volume of the EDTA rundown in cm^3			

CACULATIONS:

Weight of cement sample in 250 cm^3 = …………….(W) grams

Morality of EDTA=…………(Z) M

Volume of EDTA required react with 25 cm^3 of cement solution=………………(X) cm^3

$1cm^3$ of 1M EDTA = 56.08mg CaO (Molecular mass of calcium oxide =56.08)

Therefore X cm^3 of Z M EDTA = 56.08×10^{-3} x Xx Z g of CaO=………………………

=……………(a) grams

Therefore 250 cm^3 of cement solution contains 10 x (a) grams of CaO……………(b) grams

Percentage of CaO in cement sample = $\dfrac{b \times 100}{W}$ = …………………..

…………………%

EXPERIMENT NO: 2

Estimation of Calcium Oxide in cement by rapid EDTA method.

Aim: **To determine the percentage of calcium oxide (CaO) in a given cement sample**.

Theory: Cement contains oxides of calcium, aluminum, magnesium, silica and small quantities of iron. A known weight of the cement sample is treated with acid and the insoluble residue mainly containing silica is removed by filtration. Iron and aluminum in the filtrate are precipitated as their hydroxides and filtered. The resulting solution containing calcium and magnesium is diluted to 250 cm^3 and supplied for estimation. Patton and Reeder's indicator permits the determination of calcium in the presence of magnesium.

Calcium ions present in the solution is determined by titrating a known volume of the cement solution with EDTA. The solution is treated with diethylamine to maintain the pH at 12-14. Sodium hydroxide (4N) is added to precipitate the Mg^{2+}ion present in the solution as magnesium hydroxide and make Mg ineffective. The solution is then titrated against standard EDTA using Patton and Reeder's indicator. Glycerol is added to get a sharp end point. This indicator can be used in the estimation of calcium in the presence of magnesium as the latter is in the form of precipitate.

Ca^{2+} + Indicator → Ca - Indicator Complex
(Wine red)
Ca - Indicator Complex + EDTA → Ca – EDTA Complex + Indicator
(blue)

Procedure:

Part-A: Preparation of standard EDTA solution.

Weigh around 1.5g of disodium salt of EDTA crystals using an electronic balance and transfer the crystals carefully into 250cm^3 volumetric flask by using funnel and find the weight of the empty bottle. Add 3 cm^3 of 1:1 NH_3 and ion exchange water through the funnel to ensure that all the crystals slide down into the flask. Wash the funnel with ion exchange water and remove the funnel. Dissolve the crystals by swirling the flask Dilute with ion exchanger water and make upto the mark. Stopper and invert the flask several times so that a homogeneous solution results. Calculate the molarity of EDTA.

Part –B: Estimation of percentage of calcium oxide.

Transfer 25 cm^3 of the given cement solution into a clean conical flask using a pipette. Add 5 cm^3 of 1:1 glycerol with constant shaking of the contents of the flask followed by 5cm^3 of diethylamine (so as to bring the pH of the solution to 12.5).Add one test tube full of 4N sodium hydroxide and mix the solution well. .Add two test tube full of ion exchange water and a 2-3 drops of Patton and Reeder's indicator. Titrate against the EDTA solution till the wine red colour disappears and blue colour appears.

Note: The titration should be performed very slowly near the end point.

Result: Percentage of calcium oxide in a given cement sample is …………………%

OBSERVATION AND CALCULATIONS:

Tabular column

Trial No.	1	2	3
Final burette reading			
Initial burette reading			
Volume of $Na_2S_2O_3$ rundown in cm^3			

CALCULATIONS:

Weight of brass sample = W……………grams

Normality of sodium thiosulphate = …………..(Y) N

Volume of sodium thiosulphate required = …………(X)cm^3

1 cm^3 of 1N sodium thiosulphate = 1 milli equivalent of copper

(1 equivalent of copper = Atomic mass of copper = 63.54)

Therefore 1 cm^3 of 1N sodium thiosulphate = 0.06354 gm of copper.

X $cm^3$of'Y' N sodium thiosulphate = 0.06354 x X x Y gm of copper

= ……………………..…=………...….(a) grams

a x 10 = …………………………….= ……………………….. (b) grams

Percentage of copper in brass sample = $\frac{\text{b x 100}}{\text{W}}$ =……………………..

=……………………..%

EXPERIMENT NO: 3

Determination of percentage of copper in brass using standard Sodium thiosulphate solution

Aim: To determine the percentage of copper in a given brass sample.

Theory: Brass is an alloy of copper and zinc. It also contains small amounts of tin, lead and iron. The percentage composition of a typical brass alloy is

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

The amount of copper present in brass can be determined iodometrically. A solution of brass is made by dissolving the sample in minimum amount of nitric acid .Excess nitric acid is destroyed by boiling with urea .The solution is treated with ammonia to remove any mineral acid and acidified with acetic acid. Potassium iodide solution is added, when iodine is released due to the reduction of cupric ions and cuprous iodide is precipitated.

$$Cu + 4HNO_3 \longrightarrow Cu(NO_3)_2 + 2NO_2\uparrow \text{(Reddish brown gas)} + 2H_2O$$

$$\underset{\text{Cupric}}{2Cu^{2+}} + 4KI \longrightarrow \underset{\text{Cuprous iodide}}{Cu_2I_2\downarrow} + 4K^+ + I_2$$

The iodine liberated is titrated against sodium thiosulphate ($Na_2S_2O_3$) using starch as indicator.

$$Na_2S_2O_3 + I_2 \longrightarrow \underset{\text{(Sodium tetrathionate)}}{Na_2S_4O_6} + 2NaI$$

Starch forms a blue complex with iodine. The end point is detected by sharp disappearance of the blue colour .The volume of sodium thiosulphate required is measure of amount of copper present.

Procedure:

Part A: Preparation of brass solution.

Weigh the given brass sample accurately and transfer it into a clean 100 cm^3 beaker. Add 3cm^3 of 1:1 nitric acid and boil till the alloy dissolves completely. Add 1 test tube of ion exchange water and about 1 g of urea. Boil for about 2 minutes to destroy oxides nitrogen. Cool the mixture. Transfer the above prepared brass solution into 250 cm^3 volumetric flask, wash the beaker with ion exchange water and transfers the washing into volumetric flask. Dilute up to the mark with water and mix well.

Part B: **Estimation of percentage of copper**.

Pipette out 25 cm^3 of brass solution into a conical flask, add aqueous ammonia drop wise till a pale blue precipitate is formed. Dissolve the precipitate by adding acetic acid in drop wise. Add one test tube full of 10% KI. Titrate the liberated iodine against standard sodium thiosulphate solution till the mixture turns straw yellow. Add 10 drops of starch indicator and continue the titration till blue colour disappears and a white precipitate is left behind. Repeat the experiment for agreeing values.

Result: Percentage of copper in a given brass sample = …………………….%

OBSERVATION AND CALCULATIONS:

Weight of FAS + bottle =.......................................grams.

Weight of empty bottle= ....grams.

Weight of FAS = ……………….grams

Normality of FAS = $\dfrac{\text{Weight of FAS x 4}}{\text{Molecular Weight of FAS (392)}}$ =…………………...

=……………..(Z) N

Tabular column

Trial No.	1	2	3
Final burette reading			
Initial burette reading			
Volume of the FAS rundown in cm^3			

CACULATIONS:

Volume of FAS required (Part-A) =…………………(Y) cm^3

Blank titre value (Part B) = ………………(X) cm^3

Volume of FAS equivalent to the oxidizable impurities = (X-Y)……………...cm^3

Normality of FAS =……………. (Z) N

$1000cm^3$ of 1N FAS solution = 1 equivalent of oxygen = 8 grams of oxygen

Therefore (X-Y) cm^3 of (Z) N FAS solution = $\dfrac{8 \text{ x } (X-Y) \text{ x } Z}{1000}$ =…………………………

=……………grams of oxygen

1000 cm^3 of waste water sample = $\dfrac{8\text{x } (X-Y) \text{ x } Z}{25}$ =……………………..……

=………………grams of oxygen

COD of waste water sample = $\dfrac{8000 \text{ x } (X-Y) \text{ x } Z}{25}$ =……………….………

=………………mg of oxygen

EXPERIMENT NO: 4

Determination of chemical oxygen demand (COD) of industrial waste water

Aim: To determine the COD of a given waste water sample.

Theory: Chemical oxygen demand is a measure of the oxygen equivalent of organic and inorganic materials in a waste water sample that can be oxidized by a strong chemical oxidant. This parameter is important for industrial waste water studies. COD is expressed as mg of oxygen required to oxidize impurities present in 1000cm^3 of waste water.

The oxidizable constituents of waste water include straight chain alphatic compounds , aromatic hydrocarbons, straight chain alcohols, acids, pyridine and other oxidizable materials. straight chain compounds and acetic acid are oxidized more effectively in presence of silver ions(added silver sulphate).as catalyst. However the silver ion become ineffective in presence of halide ions (present in waste water) owing to the precipitation of silver halide. This difficulty is overcome by treating the waste water with mercuric sulphate before the analysis for COD. Mercuric sulphate binds the halide ions and make them unavailable.

A known volume of waste water sample is treated with excess of acidified potassium dichromate. Un reacted potassium dichromate is titrated with standard ferrous ammonium sulphate solution using ferrion as indicator. A blank titration is performed without the water sample. COD of the waste water sample is determined from the difference in the titre values. The method can be used to estimate COD values of 50 mg or more.

$$K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3(O)$$

$$[\ 2FeSO_4 + H_2SO_4 + (O) \longrightarrow Fe_2(SO_4)_3 + H_2O\ ] \times 3$$

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

Procedure:

Part A: Preparation of standard ferrous ammonium sulphate (FAS) solution.

Weigh out around 4.8 g of ferrous ammonium sulphate into a 250 cm^{3}volumetric flask. Add two test tube full of dilute sulphuric acid and swirl the flask to dissolve the crystals. Make up the solution to the mark with ion exchange water. Mix the solution well and calculate the normality of ferrous ammonium sulphate.

Transfer 25 cm^{3}of waste water sample into 250 cm^3 conical flask using a pipette. Add 10 cm^{3}of standard potassium dichromate solution followed by one test tube full of 1:1 sulphuric acid(containing silver sulphate and mercuric sulphate) with constant shaking of contents of the flask. Add 2-3 drops of ferroin as indicator and titrate against ferrous ammonium sulphate solution until the solution turns from blue green to reddish brown.

Part –B: Blank titration.

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

Result: COD of the given waste water sample = ……………..mg of oxygen

OBSERVATION AND CALCULATIONS:

Weight of $K_2Cr_2O_7$ + bottle =.......................................grams.

Weight of empty bottle= .... grams.

Weight of $K_2Cr_2O_7$ Crystals = ……………….grams

$$\text{Normality of } K_2Cr_2O_7 = \frac{\text{Weight of } K_2Cr_2O_7 \times 4}{\text{Molecular Weight of } K_2Cr_2O_7 \text{ (49)}}$$ =……………………

=………………(Y) N

Tabular column

Trial No.	1	2	3
Final burette reading			
Initial burette reading			
Volume of the $K_2Cr_2O_7$ rundown in cm^3			

CACULATIONS:

Weight of haematite ore in 250 cm^3 of the given solution = ……………… (W) grams

Volume of $K_2Cr_2O_7$ required =…………………(X) cm^3

$1cm^3$ of 1N $K_2Cr_2O_7$ = 1 milli equivalent of iron

$1cm^3$ of 1N $K_2Cr_2O_7$ = 0.05585 grams of iron

(1 equivalent of iron = atomic mass of iron=55.85.)

Therefore X cm^3 of (Y) N $K_2Cr_2O_7$ = 0.05585 x X x Y gram of iron = …………………..

= ………… (a) grams

$25cm^3$ of haematite solution contains (a) grams of iron.

Therefore $250cm^3$ of haematite solution contains 10 x (a) grams of iron = …………(b) grams

$$\text{Percentage of iron in the given haematite ore sample} = \frac{b \times 100}{W}$$ **=……………………**

……………….%

EXPERIMENT NO: 5

Estimation of percentage of iron in the given rust solution using standard Potassium Dichromate solution (External indicator method)

Aim: To determine the percentage of iron in haematite ore

Theory: Haematite is an important ore of iron containing ferric oxide (Fe_2O_3)and a small amount of silica (SiO_2).A known weight of the ore is digested with hydrochloric acid and the insoluble residue containing silica is removed by filtration. The filtrate is diluted to 250 $cm^3$and is supplied for the estimation.

Iron is present in the solution as Fe^{3+} (ferric) ions. It is reduced to Fe^{2+} (ferrous) ions using stannous chloride ($SnCl_2$) in hot condition in presence of concentrated HCl. The excess of stannous chloride is oxidized by treating the solution with mercuric chloride. The ferrous ions in the resulting solution is titrated against standard potassium dichromate ($K_2Cr_2O_7$) using potassium ferricyanide as external indicator. Potassium ferricyanide produces an intense deep blue colour with ferrous ions due to the formation of ferroferricyanide complex.

$$Fe_2O_3 + 6HCl \longrightarrow 2FeCl_3 + 3H_2O$$

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

$$\underset{\text{Mercuric chloride}}{SnCl_2 + 2HgCl_2} \longrightarrow \underset{\text{Mercurous chloride (Silky White ppt)}}{Hg_2Cl_2\downarrow} + SnCl_4$$

$$6Fe^{2+} \longrightarrow Fe^{3+} + e^- \times 6 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ (1)$$

$$Cr_2O_7^{2-} + 14\,H^+ + 6e^- \longrightarrow 2Cr^{3+} + 7H_2O \ldots\ldots\ldots(2)$$

Adding (1) and (2)

$$6Fe^{2+} + Cr_2O_7^{2-} + 14\,H^+ \longrightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_20$$

Procedure:

Part A: Preparation of standard Potassium dichromate solution

Weigh about 1.25 grams of $K_2Cr_2O_7$ crystals and transfer it into a 250 cm^3 of volumetric flask. Dissolve the crystals in ion exchange water, make up to the mark using water and mix well. Calculate the normality of Potassium dichromate.

Part B: Estimation of percentage of iron.

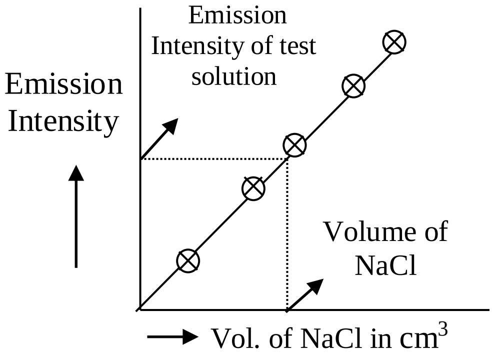
Transfer 25 cm^3 of haematite ore solution into a clean conical flask using a pipette. Add 5 cm^3 of concentrated HCl and heat the solution to boiling. Add stannous chloride to the hot solution drop wise till the yellow solution just turns colourless. Add 2 drops of excess $SnCl_2$ to ensure complete reduction. Cool, add rapidly 5 cm^3 of mercuric chloride followed by two test tube full of ion exchange water. A silky white precipitate of mercurous chlroride is formed. Place a drops of freshly prepared potassium ferricyanide as indicator on a wax paper. Add $K_2Cr_2O_7$ in the increments of 1 $cm^3$from the burette to the conical flask and mix well. Using a clean glass rod, withdraw a drop of solution from the conical flask and bring it in contact with the drop of indicator on the wax paper. The colour of the indicator turns deep blue. If blue colour develops, continue the titration by adding of Potassium dichromate solution as above till a drop of mixture fails to produce a blue colour with the indicator (use cleaned glass rod after every test). Repeat the titration with another 25 cm^3 of the haematite solution by adding of Potassium dichromate in the increments of 0.5 cm^3 from the burette.

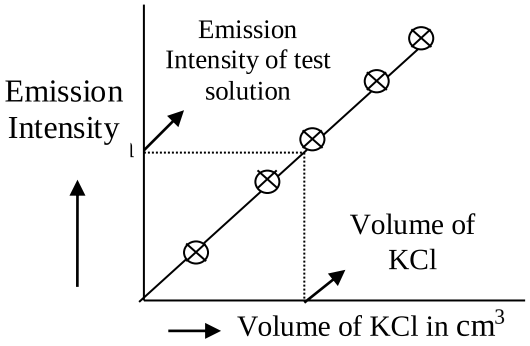
Result: Percentage of iron in a given haematite ore solution =…………………..%

OBSERVATION AND CALCULATIONS:

Volume of sodium chloride solution (cm^3)	Emission Intensity
2.0	
4.0	
6.0	
8.0	
10.0	
Test solution	

Volume of potassium chloride solution (cm^3)	Emission Intensity
2.0	
4.0	
6.0	
8.0	
10.0	
Test solution	





Demonstration Experiments

EXPERIMENT- 1

Flame photometric estimation of sodium and potassium

Aim: Estimation of sodium and potassium Flame Photometrically.

Theory: Sodium, Potassium, calcium and lithium and other common elements impart characteristic colors with the bunsen flame. The intensity of the colored flame varies with the amount of element introduced. This forms the basis of flame photometry.

When a solution containing a compound of the metal to be estimated is aspirated into a flame, the following processes occur.

i). Solvent evaporates leaving behind a solid residue.

ii). Vaporization of the solid coupled with dissociation into its constituent atoms, which are initially in the ground state.

iii). Some gaseous atoms get excited by the thermal energy of the flame to higher energy levels.

The excited atoms, which are unstable quickly, emit photons and return to lower energy state i.e. ground state. Flame photometry involves the measurement of emitted radiation.

The relationship between the ground state and excited state populations is given by the Boltzmann equation:

$$N_1/N_0 = (g_1/g_0)\, e^{\Delta E/kT}$$

N_1 = Number of atoms in the excited state; N_0 = Number of atoms in the ground state

g_1/g_0 = ratio of statistical weights for ground and excited states; ΔE = Energy of excitation = $h\nu$

k = the Boltzmann constant; T = Absolute Temperature

From the above equation, it is evident that the ratio $N_1/N_0$is dependent upon both the excitation energy E and the temperature T. An increase in temperature and a decrease in ΔE will both result in a higher value for the ratio N_1/N_0.

Procedure:

1.Transfer 2.0, 4.0, 6.0, 8.0, 10.0 cm^3 of standard NaCl solution (which is prepared by weighing accurately 1.271 g NaCl into a 1 liter standard volumetric flask and dissolving the crystals and diluting the solution up to the mark with distilled water. The solution gives
1 ppm Na/ml) into 50 ml standard volumetric flasks and dilute up to the mark with distilled water.

2. Place the distilled water in the suction capillary of the instrument and set the instrument to read zero.

3. Place each of the standard solutions in the suction capillary and set the instrument to read the flame emission intensity 2.0, 4.0, 6.0, 8.0 and10.0 respectively (rinse with distilled water between each reading) using sodium filter **(598 nm**).

4. Dilute the given test solution up to the mark with distilled water, mix well and place the solution in the suction capillary and record the reading.

5. Draw a calibration curve by plotting the emission intensity (Y-axis) and volume of NaCl solution(X-axis).

6. From the calibration curve, find out the volume of the given test solution and from which calculate the amount of Na in the water sample.
7. Using the above procedure the amount of potassium in KCl Solution can be estimated.

Result: The amount of sodium and potassium present in the given sample of water

=………………..mg

EXPERIMENT- 2

Synthesis of ZnO nano particle by Precipitation method

Materials

Zinc nitrate as the precursor, KOH as a precipitating agent to synthesize ZnO nanoparticles

Preparation

ZnO nanoparticles can be synthesized by direct precipitation method using zinc nitrate or zinc chloride and KOH or NaOH. The aqueous solution (0.2 M) of zinc nitrate ($Zn(NO_3)_2.6H_2O$) and the solution (0.4 M) of KOH to be prepared with deionized water, respectively. The KOH solution should be added slowly into zinc nitrate solution(taken in a beaker) at room temperature under vigorous stirring, which resulted in the formation of a white suspension. The white product formed should be filtered and washed three times with distilled water, and once with absolute alcohol. The obtained product to be calcined at 180 °C in hot air oven for 2 hr. The prepared ZnO nano particle to be fine powder using mortar & pestle.

Note: Any metal chlorides, nitrates & sulphates can be used as precursors for the preparation of nano particles.

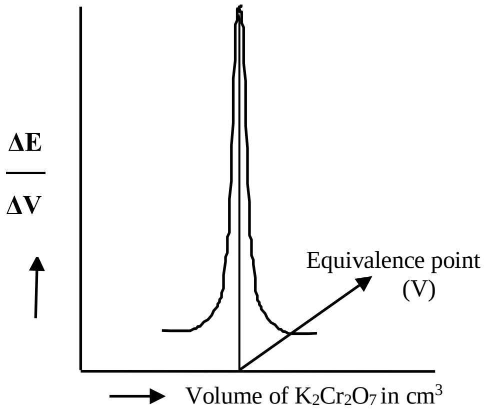
MODEL PROCEDURES

MODEL PROCEDURES
Instrumental experiments

1. Potentiometric estimation of FAS using standard K2Cr2O7 solution.

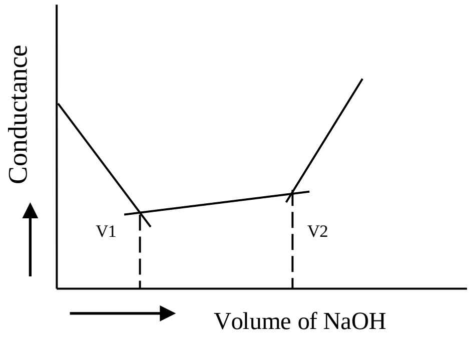
Pipette out $25cm^3$ ferrous ammonium sulphate into a beaker. Add 2 test tube of dilH_2SO_4. Immerse platinum electrode-calomel electrode assembly into it. Connect the electrode assembly to a potentiometer and measure the potential. Add $K_2Cr_2O_7$ from the burette in the increments of $0.5cm^3$ and measure the potential after each addition.

Plot a graph of $\Delta E/ \Delta V$ against volume of $K_2Cr_2O_7$ and determine the equivalence point. Using the normality of $K_2Cr_2O_7$, calculate the normality and the weight of FAS in the given solution.



2. Conductometric estimation of the acid mixture.

Pipette out $50cm^3$ of the acid mixture into a clean $100cm^3$beaker. Immerse the conductivity cell into the acid mixture. Connect the cell to a conductivity meter and measure the conductance. Add NaOH from a burette in increments of 0.5 cm^3 and measure the conductance after each addition. Plot a graph of conductance against volume of NaOH added. Determine the neutralization points (V_1 and V_2) from the graph and calculate the normality and the amount of HCl & CH_3COOH in one litre.



3. Determination of viscosity co-efficient of a liquid using Ostwald's viscometer.

Clean the viscometer with water, rinse with acetone and dry it. Pipette out $10cm^3$ of water into a wide limb of the viscometer and suck the water through the other limb. Determine the time of flow between two fixed points one above and one below the bulb in the narrow limb of the viscometer. Repeat the procedure and calculate the average time of flow of water (t_1). Pour out the water, wash the viscometer, rinse with acetone and dry it. Now pipette out $10cm^3$ of liquid into the wider limb and determine the average time of flow for liquid as before. Using the density of the liquid and water and the viscosity co-efficient of water, calculate the viscosity co-efficient of the given liquid.

The viscosity co-efficient of a given liquid is given by,

$$\eta_1/\ \eta_2 = t_1d_1/t_2d_2$$

$$\eta_2 = t_2d_2 \text{ x } \eta_1/t_1d_1$$

Where,

η_1= Viscosity co-efficient of water in milli poise

η_2= Viscosity co-efficient of liquid in milli poise

d_1 = Density of water in gm/cm^3

d_2 = Density of liquid in gm/cm^3

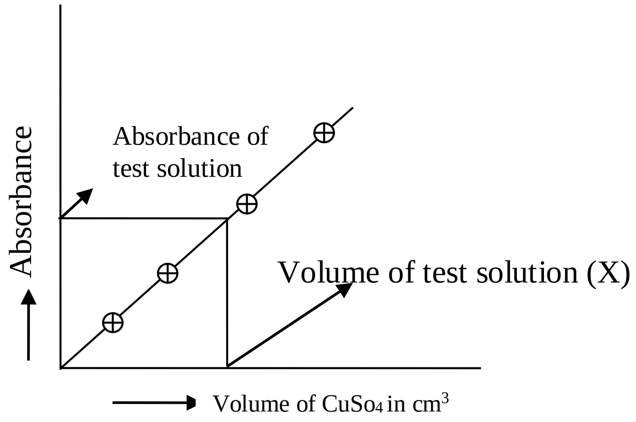
t_1 = Time flow of water in seconds

t_2 = Time flow of liquid in seconds

Viscosity co-efficient of the given liquid = η_2 = …………………...milli poise

4. Colorimetric estimation of Copper.

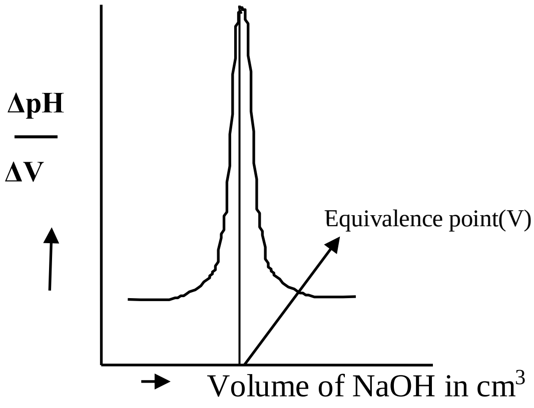
Transfer 2.5, 5.0, 7.5, 10.0 and $12.5cm^3$ of copper sulphate solution into a separate $25cm^3$ volumetric flasks. Add $2.5cm^3$ of ammonia to each one of them and also into the test solution. Dilute up to the mark with water and mix well. Measure the absorbance of each of the solution against blank solution (only ammonia and water) at 620nm. Plot a graph of absorbance against volume of copper sulphate. From the graph find out the volume of test solution (copper sulphate) and calculate the amount of copper in a given test solution.

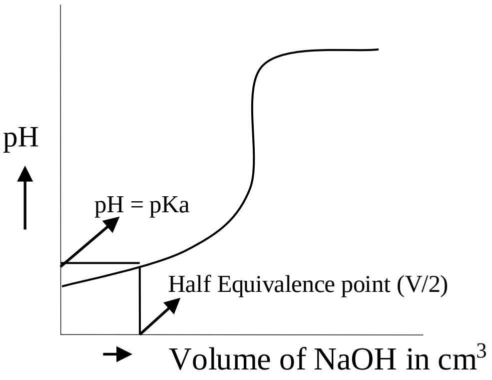


5. Determination of pKa of weak acid using pH meter.

Pipette out 25cm^3 of the weak acid into a beaker. Immerse the glass electrode and calomel electrode assembly into it. Connect the electrodes to P^H meter and measure the pH of acid. Now add NaOH from the burette in increments of 0.5cm^3 and measure the pH after each addition.

Plot a graph of ΔpH/ ΔV against volume of NaOH and determine equivalence point. Plot another graph, pH against volume of NaOH and determine the half equivalence point and pKa value of the given weak acid.





Volumetric experiments

1) Estimation of Total hardness of water by EDTA complexometric method.

Part-A: **Preparation of standard EDTA solution.**

Weigh out given EDTA crystals accurately into 250cm^3 volumetric flask. Add 3cm^3 of 1:1 $NH_3$and dissolve the crystals, dilute up to the mark with distilled water and mix well.

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

Part-B: Determination of Total hardness of water sample.

Burette: Standard EDTA solution.
Conical Flask: 25cm^3 of hard water sample + 3cm^3 of NH_3–NH_4Cl (Buffer solution) (to maintain pH-10)
Indicator: Eriochrome Black-T
End Point: Disappearance of wine red colour (Appearance of blue colour).

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

2) Estimation of CaO in cement by rapid EDTA method.

Part-A: Preparation of standard EDTA solution.

Weigh out given EDTA crystals accurately into 250cm^3 volumetric flask. Add 3cm^3 of 1:1 $NH_3$and dissolve the crystals and dilute up to the mark with distilled water. Mix well for uniform concentration.

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

Part-B: Estimation of percentage of CaO.

Burette:	Standard EDTA solution.
Conical Flask:	25cm^3 of cement solution +5cm^3 of glycerol+ 5cm^3 of diethylamine + 1 t.t 4N NaOH + 2 t.t H_2O
Indicator:	Patton and Reeder
End Point:	Disappearance of wine red colour (Appearance of blue colour).

From the volume of EDTA consumed calculate the percentage of CaO in cement sample.

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

Part A: Preparation of Brass solution.

Weigh out the given brass sample into a clean 100cm^3 beaker. Add 3cm^3 1:1HNO_3. Boil till all brown fumes are expelled. Add 1 t.t of distilled water and one spatula urea, boil for 2 minutes. Cool to room temperature and transfer the solution into 250 cm^{3}volumetric flask, makeup up to the mark with ion exchange water and mix well for uniform concentration.

Part B: Estimation of percentage of copper in brass.

Burette:	Standard $Na_2S_2O_3$ Solution.
Conical Flask:	25cm^{3}ofBrass Solution + Add NH_4OH till slight ppt.+ Add Dil CH_3COOH to dissolve the ppt+ Add1 t.t 10% KI
Indicator:	Starch added towards the end point
Colour change:	Disappearance of blue colour (White ppt left).

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

4) Determination of Chemical Oxygen Demand (COD) of industrial waste water.

Part-A: **Preparation of standard FAS solution**.

Weigh out the given ferrous ammonium sulphate (FAS) crystals accurately into a 250cm^3 volumetric flask. Add 2 t.t dil H_2SO_4, dissolve the crystals, dilute up to the mark with water and mix well for uniform concentration.

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

Part-B: Determination of COD of waste water sample.

Burette: Standard FAS solution.
Conical Flask: 25cm^3 of waste water sample +10cm^3 of $K_2Cr_2O_7$ solution +1 t.t of 1:1 H_2SO_4.(containing Ag_2SO_4 and $HgSO_4$)
Indicator: Ferroin
End Point: Blue green to reddish brown.

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

5) Estimation of percentage of iron in the given rust solution using standard Potassium Dichromate solution (External indicator method)

Part-A: **Preparation of standard potassium dichromate solution.**

Weigh out the given potassium dichromate crystals accurately into a 250cm^3 volumetric flask, dissolve in distilled water and dilute up to the mark with water. Mix well for uniform concentration.

$$\text{Normality of Potassium dichromate} = \frac{\text{Weight of potassium dichromate x 4}}{\text{Equivalent weight of potassium dichromate (49)}}$$

Part-B: Estimation of percentage of Iron.

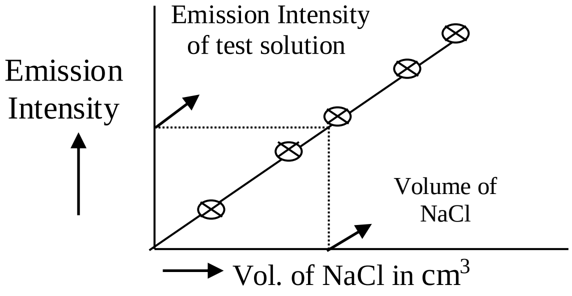
Burette: Standard $K_2Cr_2O_7$ Solution.
Conical Flask: 25cm^3 of haematite ore solution + ¼ t.t Conc. HCl, heat+ $Sncl_2$ drop wise till colorless. Cool and add ¼ t.t of mercuric chloride
Indicator : $K_3Fe(CN)_6$ (potassium ferricyanide) external indicator.
Colour change: A drop of test solution fails to produce blue colour with indicator.

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

MODEL PROCEDURES:
Demonstration experiment

1. Flame photometric estimation of sodium and potassium

Transfer 2.0, 4.0, 6.0, 8.0, 10.0 cm^3 of standard NaCl or KCl solution into $50cm^3$ standard volumetric flasks and dilute up to the mark with distilled water. Set the instrument to read zero with distilled water. Place each of the standard solutions in the suction capillary and set the instrument to read emission intensity (rinse the beaker with distilled water for each reading). Dilute the given test solution up to the mark with water and mix well and place the solution in the suction capillary and record the reading. Draw a calibration curve by plotting the emission intensity against volume of NaCl of KCl solution. From the calibration curve, find out the volume of the given test solution and calculate the amount of sodium and potassium.



EXPERIMENT- 2

2. Synthesis of ZnO nano particle by Precipitation method

ZnO nanoparticles can be synthesized by direct precipitation method using zinc nitrate or zinc chloride and KOH or NaOH. The aqueous solution (0.2 M) of zinc nitrate ($Zn(NO_3)_2.6H_2O$) and the solution (0.4 M) of KOH to be prepared with deionized water, respectively. The KOH solution should be added slowly into zinc nitrate solution(taken in a beaker) at room temperature under vigorous stirring, which resulted in the formation of a white suspension. The white product formed should be filtered and washed three times with distilled water, and once with absolute alcohol. The obtained product to be calcined at 180 °C in hot air oven for 2 hr. The prepared ZnO nano particle to be fine powder using mortar & pestle.

VIVA-VOCE QUESTIONS AND ANSWERS

VIVA- VOCE QUESTIONS AND ANSWERS

Some Basic questions:

1) **Define Normality (N)**
   It is the no. of gram equivalents of the solute dissolved in one dm^3 of the solvent.
2) **Define Molarity (M)**
   It is the number of moles of the solute dissolved in one dm^3 of the solvent.
3) **Define Molality (m).**
   It is the number of moles of the solute dissolved in one kg of the solvent.
4) **What are indicators?**
   Indicators are the substances, which indicate the end point in a titration by changing its colour.
5) **What are different types of indicators used in volumetric analysis?**
   1. Acid base indicators, e.g. Phenolphthalein.
   2. Metal ion indicators, eg. Eriochrome black-T, Patton and Reader's
   3. Redox indicator, eg, Ferroin
   4. Internal indicator eg, phenolphthalein, Eriochrome black-T, Patton and
      i. Reader's etc.,
   5. External indicator, eg, potassium ferricyanide.
   6. Self indicator, eg, Potassium permanganate.
6) **What is buffer?**
   Buffer solution is substance, which resist change in pH value on adding small quantities of acid or base to them..Ex, CH_3COOH & CH_3COONa, NH_4OH & NH_4Cl
7) **What is a standard solution?**
   A standard solution is one whose concentration is known.
8) **What is oxidation?**
   Oxidation is a chemical reaction involving removal of hydrogen or electrons or addition of oxygen.
9) **What is reduction?**
   Reduction is a chemical reaction involving removal of oxygen or addition of hydrogen or electrons
10) **What is redox titration?**
    A reducing agent is titrated against an oxidizing agent or vice versa. Redox titration includes oxidation of one reactant and reduction of the other.
11) **What is complexometric titration?**
    A titration, which involves the formation of complex salts .ex; EDTA vs. hard water.
12) **What is an acid?**
    A molecule or other species which can donate a proton or accept an electron pair in reactions.
13) **What is base?**
    A base is a chemical species that donates electrons or hydroxide ions or that accepts protons.
14) **What is an oxidizing agent?**
    A substance that tends to bring about oxidation by being reduced and gaining electrons. Eg: KM_nO_4, $K_2Cr_2O_7$, KNO_3, HNO_3, Halogens etc..

15) What is reducing agent?

A substance that tends to bring about reduction by being oxidized and losing electrons.
Eg: $LiAlH_4$, $NaBH_4$, $Na_2S_2O_3$, HCOOH etc.

Instrumental experiments

Experiment No.1

Potentiometric estimation of FAS using standard $K_2Cr_2O_7$ solution.

1) What is Potentiometric titration?

The determination of the equivalent point of redox titration on the basis of potential measurement is called a Potentiometric titration.

2) What are the electrodes used in the determination of FAS potentiometrically?

The indicator electrode used is the platinum electrode (acts as anode) and the reference electrode used is the calomel electrode (acts as cathode).

3) Give the principle of Potentiometric titrations.

Potentiometric titration is a technique by which change in concentration of the ions in redox (reduction-oxidation) reaction is determined with a change of electrode potential. The EMF of the cell consisting of indicatior electrode (platinum) and the reference electrode (calomel) is measured while the titration is in progress. The end point is marked by a sudden rise in potential.

4) What is an indicator electrode?

The electrode whose potential is dependent upon concentration of the ions which is to be determined in solution is termed as indicator electrode. eg: platinum electrode .

5) Which equation is used in calculating electrode potential?

Nernst equation.

$$E = E^o + \frac{0.0591}{n} \log [Mn^+]$$

6) What is the reaction between FAS and potassium dichromate?

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

7) What determines the potential in the redox reaction FAS and dichromate?

Ratio of concentrations of the oxidized and reduced forms i.e., $(Fe^{3+}) / (Fe^{2+})$

8) Why is H_2SO_4 added to FAS solution during emf measurement?

Reaction between FAS and $K_2Cr_2O_7$ is a redox reaction, where oxidizing agent $K_2Cr_2O_7$ only in the presence of acidic medium oxidizes Fe^{2+} to Fe^{3+}.

9) Why the emf is rises steeply after the equivalent point?

This is because, the potential of the solution before the equivalence point is determined $Fe^{2+} \rightarrow Fe^{3+}$ by system only i.e., 0.75V, while at equivalence point, it is determined by both Fe^{3+} and $Cr_2O_7^{2-}$ ions which is = 1.04V. But beyond equivalence point, the potential of the solution is determined by $Cr_2O_7^{2-} / Cr^{3+}$ only i.e. = 1.33V. Therefore, just after the equivalence point, the potential of the solution raises steely.

10) What are the advantages of Potentiometric titration?

Turbid and mixture of very dilute solutions can be titrated. Results are very accurate because end point is obtained by graphical method.

Experiment No.2

Conductometric estimation of the acid mixture

1) **What is conductance?**

The reciprocal of resistance is called conductance

2) **What is conductivity?**

The reciprocal of resistivity is called conductivity.

3) **What is the unit of conductance?**

The unit of conductance is ohm^{-1}or S.

4) **State ohm's law.**

Ohm's law states that the current, I (ampere), flowing in a conductor is directly proportional to the applied emf, E (volt) and inversely proportional to the resistance, R (ohm) of the conductor.

5) **Mention the different types of conductance?**

A solution may have the following conductance.

   a) Specific conductance
   b) Equivalent conductance.
   c) Molar conductance.

6) **Which of the above conductance measured during conductometric titration?**

The specific conductance is measured.

7) **What is specific conductance?**

It is the conductance of a solution placed between two platinum electrodes of $1cm^2$. area and kept 1 cm apart.

8) **What is equivalent conductance?**

It is the conductance of the solution, which contains 1-gram equivalent of solute, when placed between two electrodes of 1 cm apart.

9) **What is molar conductance?**

It is the conductance of a solution, which contains I gram molecular weight of a substance, when placed between two electrodes of 1 cm apart.

10) **What is the principle of conductometric titration?**

In an acid base titration, there is sudden change in the conductance, of the solution near the neutralization (or equivalence) point and the neutralization point is determined graphically by plotting conductivity against volume of base.

11) **What factor determines the conductance of a solution?**

Two factors determining the conductance of a solution are

   a. . Mobility of ions- higher the mobility, higher is the conductance and
   b. Number of ions- more the number of ions in solution more is the conductance.

12) **What is the unit of conductivity?**

$Ohm^{-1}cm^{-1}$ or $ohm^{-1}m^{-1}$.

13) **In the titration of HCl with a strong base (NaOH), the conductance decreases then increases steeply. Why?**

Upon adding a strong base to a strong acid, the conductance falls due to the replacement of highly mobile H^+ ions of the strong acid (HCl) by less mobile Na^+ ions of the base. The conductance falls till all the H^+ ions are replaced (i.e. till HCl is neutralized completely). The conductance then rises steadily as the weak acid (CH_3COOH) is converted into its salt. Finally, the conductance rises steeply as excess of alkali (OH^- ions) is introduced, i.e., due to highly mobile OH^- ions.

14) **What are the advantages of conductometric titrations?**

It is very accurate in dilute as well as in concentrated solutions. It can be used for colored solutions and mixture of acids

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Experiment No.3

Determination of viscosity co-efficient of a given liquid using Ostwald's viscometer.

1) **What is viscosity?**

The property of a liquid exhibiting resistance to flow is known as viscosity. It is the property which opposes the relative motion of adjacent layer of the liquid due to internal friction.

2) **What is viscosity coefficient of a liquid?**

It is defined as the tangential force per unit area ($dynes/cm^2$) required to maintain a unit velocity gradient between two successive layers of liquid situated unit distance apart.

3) **What is density of liquid?**

The density of a liquid is its mass/ its volume.

4) **How does viscosity vary with temperature?**

Viscosity of a liquid decreases with increase in temperature.

5) **What are the factors that influence the viscosity of a liquid?**

   a. Increase in molecular weight results in an increase in viscosity.
   b. Branched chain compounds have higher viscosity than those involving straight chain compounds.
   c. The polar compounds are more viscous than the polar ones. The presence of hydrogen bonds causes the viscosity to increase.
   d. Temperature has marked influences by the viscosity of a liquid.

6) **What are the factors which affect the viscosity of liquid?**

Area of the velocity gradient, volume and temperature.

7) **What is the law base on the viscous flow of liquid through capillary tubes?**

The law based in the viscous flow of liquid through capillary tubes Poiseuilles law. It is expressed as $\eta = \frac{\pi p r^4 t}{8vl}$

Where η = Viscosity co-efficient; p = Hydrostatic pressure; r = Radius of the tubes; t = Time required for the volume; v = of the liquid to flow through the tube of the length, l.

8) **What is the unit of viscosity coefficient?**

Milli poise, SI unit being Nsm^{-2}

9) **Write the expression used to calculate viscosity coefficient of liquid**

$\eta_1/\eta_2 = t_1d_1/t_2d_2$

$\eta_2 = t_2d_2 \text{ x } \eta_1/t_1d_1$

Where,

η_1 = Viscosity co-efficient of water in milli poise
d_1 = Density of water in gm/cm^3
d_2 = Density of liquid in gm/cm^3
t_1 = Time flow of water in seconds
t_2= Time flow of liquid in seconds

10) **Why should the viscometer be dried before the measurements are done?**
The viscometer should be dried to avoid formation of an emulsion, which changes the rate of flow of the liquid.

11) **Why do require laboratory temperature for viscosity determination?**
Because, the physical constants like density and viscosity of a liquid vary with temperature.

12) **Why is viscometer dipped in water bath?**
Viscometer is dipped in water bath to maintain constant temperature

13) **Why acetone used for cleaning viscometer?**
Acetone is a volatile organic liquid. To dry the viscometer quickly, it is rinsed with acetone.

Experiment No.4

Colorimetric estimation of Copper.

1) **What is colorimetry?**
It is study of variation in the intensity of a given coloured solution with the change in the concentration of a given coloured component in the visible region.

2) **What is the range of wavelengths of light in the visible region?**
400 to 700 nm.

3) **What is the basic principle of colorimetric measurements?**
It consists of comparing under well defined conditions, the colour produce by the substance in unknown amount with the same colour produced by a known amount of the material being determined.

4) **State Beer's law.**
The intensity of a beam of monochromatic light decreases exponentially as the concentration of the absorbing substance increases arithmetically.

5) **State Lamberts law.**
The intensity of the transmitted light decreases exponentially as the thickness of the absorption medium increases arithmetically.

6) **State Beer- Lamberts law.**
The amount of light absorbed is directly proportional to the concentration (c) of the solution and directly proportional to the path length (l)

$A= \epsilon cl$

Where, ϵ = molar extinction coefficient.

7) What is the relation between absorbance and concentration?

$A = \varepsilon ct$; If t is kept constant, $A \alpha C$.

8) What is blank solution?

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

9) Why ammonia is added to copper sulphate solution in colorimetry?

Ammonia is added to $CuSO_4$ to form a cuprammonium complex with cupric ions which has a deep blue colour

10) **Why is blank solution used in colorimetric estimation?**

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

11) Why is colorimetric determination of copper done at a fixed with wave length of 620 nm?

The absorbance is measured at 620 nm since the cuprammonium complex $[Cu(NH_3)_4]^{2+}$ shows maximum absorbance at this wave length.

12) What is a calibration curve? What is its usefulness?

A plot of absorbance against concentration of series of standard solutions containing ions under test gives a straight line passing through the origin. Knowing the absorbance of the test solution, its concentration can be determined from the calibration curve.

13) What is the complex formed when ammonia solution is added to the $CuSO_4$ solution.

Cuprammoniumsulphate$[Cu(NH_3)]SO_4$

Experiment No.5

Determination of pKa of a weak acid using pH meter.

1) What is a weak acid?

A weak acid is an acid, which ionizes to a small extent in solution.

2) Give an example for a weak acid?

Acetic acid CH_3COOH, Formic acid HCOOH.

2) What are the ions formed by the dissociation of acetic acid?

Dissociation of acetic acid gives acetate (CH_3COO^-) ions and hydrogen ions (H^+)

$$CH_3COOH \longrightarrow CH_3COO^- + H^+$$

1) What are strong acids?

A strong acid is an acid, which ionizes completely in solution

5) Give example for strong acids

Strong acids: HCl, HNO_3, and H_2SO_4

6) What is pH of a solution?

pH is a measure of hydrogen ion concentration.

$pH = -\log [H^+]$

7) What are the pH values of acidic, neutral alkaline solutions?

For acidic solutions pH is between 0 and 7, for neutral 7 and for alkaline solutions between 7 & 14.

8) What is Ka?

Ka is the dissociation constant of weak acid. It is given by the equation

$$Ka = \frac{[CH_3COO^-]\,[H^+]}{[CH_3COOH]}$$

9) What is pKa of weak acid?

pKa of weak acid is the negative logarithm to base 10 of dissociation constant of the weak acid . pKa = - $\log_{10}Ka$

10) How are pH and pKa related?

Henderson equation; pH =pKa + log [Salt] / [acid]

11) What is the significance of Ka and pKa? OR How pKa and Strength the acid related?

Smaller the value of Ka weaker will be the acid vice versa. Greater the value of the pKa, weaker is the acid and vice versa.

12) What are the electrodes used in pH determination?

A combination of glass and calomel electrode. Glass electrode is an ion selective electrode which responds only to H^+ and develops a potential. Glass electrode is the indicator electrode and calomel electrode is the reference electrode.

12) What is reference electrode?

Electrode whose potential is known(standard electrode potential) which is used to determine the emf of an unknown electrode.

Eg: Calomel electrode.

12) What is Ion selective electrode?

Ion selective electrodes are membrane electrodes that respond selectively to ions in the presence of others.

Eg: Glass electrode.

13) How pH becomes equal to pKa at half equivalence point?

At half equivalence point molar concentration of salt is equal to molar concentration of the acid i.e. (salt) = (acid)

Therefore log (salt)/(acid) = log1= 0

Thus pH = pKa + 0 = pKa

14) How pH measurement is made?

By determining the EMF of the cell containing glass electrode and calomel electrode in the test solution.

15) Why pH increases suddenly after the equivalence point?

At the end point all the weak acid has been neutralized by the base into salt. Afterwards the concentration of hydroxyl ions increases resulting in sudden increase in pH.

Volumetric experiments

Experiment No.1

Estimation of Total hardness of water by EDTA complexometric method.

1) **What is hard water?**

   Hard water is the water which does not produce lather easily with soap and also produces scale in hot water pipes, heaters, boilers, etc.

2) **How is hardness of water caused?**

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

3) **How is hardness in water classified?**

   Hardness of water is classified as i) Temporary hardness and ii) Permanent hardness.

4) **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.

5) **How is temporary hardness removed?**
The temporary hardness of water can be removed by simple boiling water during which bicarbonates decompose to give insoluble carbonates.

$$Ca(HCO_3)_2 \xrightarrow{\Delta} CaCO_3 \downarrow + H_2O + CO_2$$

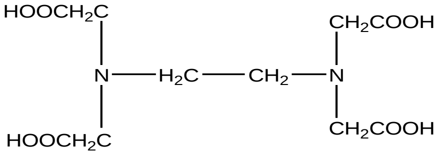
6) **How is permanent hardness removed?**
Permanent hardness can be removed by the following methods
   a) Reverse osmosis
   b) Permutite process
   c) Zeolite process

7) **What do you mean by total hardness water?**
The sum of the both temporary and permanent hardness is called the total hardness of water.

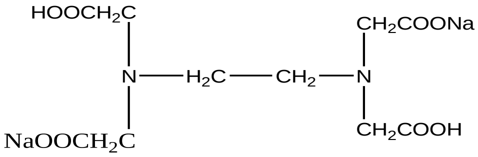
8) **How do express the total hardness of water?**
Total hardness is expressed as part per million (ppm) of $CaCO_3$.

9) **What is the full form of EDTA?**
EDTA is Ethylene diamine tetra acetic acid.

10) **Give the structure of EDTA.**



EDTA



Disodium salt of EDTA

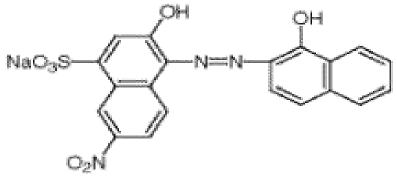
11) **Why is disodium salt of EDTA preferred to EDTA?**
EDTA is sparingly soluble in water. Its disodium salt is more soluble as it is ionisable and hence it is preferred.

12) **Why is ammonia solution added while preparing EDTA solution?**
Ammonia solution is added during the preparation of EDTA solution to increase the rate of dissolution of the salt.

13) **What is buffer solution?**
The solution which resists changing in pH value even after adding small amount of an acid or a base to it, is called a buffer solution.

14) **Why is ammonia-ammonium chloride buffer added?**
Ammonia-ammonium chloride buffer is added to maintain a pH of 10, the desired pH for the titration. Otherwise, pH decreases as H^+ ions are released due to the substitution of metal ions for H^+ ions in EDTA.

15) **What is the chemical name of Eriochrome Black T?**
The chemical name of Eriochrome Black-T is sodium (1- hydroxyl-2-naphthylazo)-6-nitro-2-naohthol-4-sulphaonate, SHNNNS).



16) **Why is the indicator Eriochrome Black-T (EBT) shows wine red colour at the beginning and blue colour at the titration?**

When a small amount of Eriochrome Black-T, which is blue in colour, is added to a hard water with a pH of about 10, it combines with a few calcium and magnesium ions to form a weak complex which is wine red in colour as shown in the below.

$$\underset{\text{Blue}}{M^{n+} + EBT} \rightarrow \underset{\text{wine red complex}}{M\text{-}EBT}$$

During the titration with EDTA, all free hardness ions are complexed according to the equation,

$$M + EBT + EDTA \rightarrow \underset{\text{Stable complex}}{M\text{-}EDTA} + \underset{\text{blue}}{EBT}$$

This action frees the Eriochrome Black-T indicator from the complex, (M-EDTA), and the wine red colour changes to a blue colour at the end of the titration.

17) **What are harmful effects of hard water?**

Hard water causes the scale formation in boilers there by leading to its explosion. Hard water is not suitable for washing.

18) **What is the application of hardness data in environmental engineering practice?**

a). Hardness of water is an important consideration in determining the suitability of water for domestic and industrial uses.

b). Determination of hardness serves as a basic for routine control of softening processes.

Experiment No.2

Estimation of CaO in cement by rapid EDTA method.

1) What is cement?

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

2) What are constituents of cement ?

Cement contains CaO, SiO_2, Al_2O_3, MgO etc.

3) What is the prime constituent of cement?

Calcium oxide is the prime constituent of cement, because the quality of cement depends on % of CaO.

4) How cement solution is prepared?

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

5) What is the function of diethyl amine?

It maintains the pH of the reaction mixture at 12.5.

6) What is role of glycerol?

Glycerol is added to get sharp end point.

7) What is the function of NaOH?

NaOH is added to precipitate Mg as $Mg(OH)_2$ and there by Mg becomes ineffective.

8) Which indicator is used in determination of CaO in cement solution? And why?

Patton and Reeder’s indicator is used in the determination of CaO in cement solution as this indicator gives a sharp colour changes in the pH ranges 12 - 14.

9) Why Eriochrome Black-T indicator is cannot be used in this experiment?

1. Eriochrome Black-T indicator cannot be used in this experiment, because, it forms a very weak complex with calcium ions in the pH range 10-11 only.
2. EBT used to determine both Ca& Mg (Total hardness). Whereas Patton and Reeder's indicator only for Calcium.

10) Why this titration is called Rapid EDTA method?

The titration is called rapid EDTA method because, by this method, Ca^{2+} ions in cement solution are estimated directly and quickly by using standard EDTA solution without removing the other metal ions.

Experiment No.3

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

1) What are the constituents of the brass?

Brass is an alloy of Cu (60 – 80%) and Zn (20 – 40%) with small amount of Sn, Pb and Fe.

2) What is an alloy?

An alloy is a homogeneous mixture of two or more metals.

3) How is brass solution is prepared?

Brass solution is prepared by dissolving the brass sample in minimum amount of conc.HNO_3. Excess HNO_3 is destroyed by boiling with urea.

4) What is the purpose of adding the urea in copper experiment?

To destroy the excess HNO_3 and completely remove the oxides of nitrogen.

5) What is the role of NH_4OH?

NH_4OH is added to neutralize excess of HNO_3.

6) What is the need to neutralize the HNO_3?

The mineral acid, HNO_3 is a powerful oxidizing agent. It also oxidizes KI to I_2. Therefore the amount of I_2 liberated does not correspond to the exact concentration of Cu^{2+} ions and hence erroneous result.

7) What is iodometric titration?

Redox titration using sodium thiosulfate, as a reducing agent is known as iodometric titration since it is used specifically to titrate iodine. The iodometric titration is a general method to determine the concentration of an oxidising agent in solution.

8)What is starch?

Starch is a polysaccharide with general formula $(C_6H_{10}O_5)_n$

9) Why is potassium iodide added to the brass solution although copper in brass is determined?

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

10) Why is the starch indicator added towards the end of the titration?

Starch forms a water insoluble blue complex with iodine which makes the detection of end point difficult. Hence it is added near the end point when the concentration of iodine is low.

11) Why brass solution turns brown upon addition of KI ?

Due to liberation of iodine

12) What is the white precipitate left behind at the end point?

Cuprous iodide (Cu_2I_2).

13) Why acetic acid is added?

To neutralize excess of ammonium hydroxide and to make the solution slightly acidic.

14) How the liberated iodine is estimated?

The liberated iodine is estimated by titrating against standard sodiumthiosulphate solution using starch indicator. Iodine reacts with sodium thiosulphate to form sodium iodide and sodium tetrathionate.

Experiment No.4

Determination of COD of industrial waste water

1) What is meant by industrial waste water?

The waste water coming out from the industrial establishments such as chemical plants, fertilizer industries, leather tanneries, sugar and paper industries, bewereries, textiles mills, oil refineries, pharmaceutical units is called as industrial sewage.

2) What is chemical oxygen demand, (COD)?

COD is the amount of oxygen required to oxidize completely organic and inorganic matter present in one litre of waste water sample using strong oxidizing agent such as acidified $K_2Cr_2O_7$ solution.

3) **What is biological oxygen demand, (BOD)?**

BOD is the amount of dissolved oxygen consumed by the micro organisms for the biological oxidation of the organic matter in one litre of waste water under aerobic conditions at 20ºC for a period of five days.

3) **What is another name for FAS?**

FAS can also be called as Mohr’s salt. [(NH4)2Fe(SO4)2·6H2O]

4) Why sulpuric acid is added during the preparation of a standard solution of FAS?

To prevent the hydrolysis of ferrous ions.

5) What is the significance of COD?

COD is importance for water treatment plant & industrial waste water studies.

6) What is the difference between COD & BOD?

COD is measure of oxygen demand for organic and inorganic matter where as BOD represents the oxygen demand for only biologically oxidizable organic matter.

7) Name the pollutants present in waste water?

Straight chain aliphatic hydrocarbons, aromatic hydrocarbons, pyidine etc.

8) What is the function of silver sulphate?

Silver sulphate acts as the catalyst for the oxidation reaction of organic compounds like straight chain aliphatichydrocarbons, aromatic hydrocarbons, pyidine etc.,

9) What is the role of mercuric sulphate in the determination of COD?

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

10) What is the indicator used in COD experiment and what is the colour change at endpoint?

Ferroin indicator & bluish green to reddish brown.

11) What is back titration?

Back titration is generally performed in the sample does not react with the standard solution. It is performed by taking excess of another reagent which can react with the sample and the standard solution.

12) What is blank titration?

Since the back titration gives the amount of reagent left in excess and not the amount reagent used up by the sample, blank titration is performed in the same way as the back titration but without using the sample. Difference between the blank and back titre values gives the amount of reagent used up.

13) What are the products formed on oxidation of organic matter?

$K_2Cr_2O_7$ oxidizes organic matter to CO_2 and H_2O

14) What is the unit in which COD is expressed?

COD is expressed in mg of oxygen per dm^3 of waste water.

15) What are the oxidizable impurities present in waste water?

Wastewater contains organic impurities such as straight chain aliphatic hydrocarbons, straight chain hydrocarbons, alcohols, aromatic etc.

Experiment No.5

Estimation percentage of iron in the given rust solution using standard Potassium Dichromate solution (external indicator method).

1) What is an ore?

Naturally occuring mineral from which metal can be extracted economically is called ore.

2) What is mineral?

A mineral is a naturally occurring mettalic compound.

3) What are the main constituent of haematite ore?

The main constituents of haematite ore is ferric oxide (Fe_2O_3) & small amount of silica.

5) What is the role of stannous chloride ($SnCl_2$) in the determination of iron in haematite ore?

Stannous chloride acts as a reducing agent. It reduces Fe^{3+} to Fe^{2+} i.e. ferric to ferrous.

5) What is the role of $HgCl_2$?

To remove the excess of $SnCl_2$. When $HgCl_2$ added, excess of $SnCl_2$ is oxidized to stannic chloride ($SnCl_4$) and a silky white precipitate of mercurous chloride is obtained.

6) What happens if excess $SnCl_2$ is not removed? or Sometimes instead of a silky white ppt we get a black (grey) ppt after adding $HgCl_2$ .why ?

Excess $SnCl_2$ reduces $HgCl_2$ to a grey ppt. of Hg. Formation of grey ppt. indicates that an excess of $SnCl_2$ has been added and in such a case that trial is rejected.

7) Why potassium ferricyanide is not used as an internal indicator in the estimation of iron by $K_2Cr_2O_7$?

Because it forms a blue complex of ferroferricyanide with reaction mixture. Therefore end point can not be detected.

8) **What is the colour of potassium ferricyanide indicator with ferrous ions?**

Deep blue colour due to the complex of ferriferrocyanide.

9) **How do you calculate the equivalent weight of dichromate?**

Since 6 e^- are involved in the reduction of dichromate ions to chromic ions, the equivalent weight of dichromate is one 6th of its molecular weight.

10) **Why the colour of indicator drop remains the same at the end point?**

$K_3[Fe(CN)_6]$ does not impart blue colour because there is no ferrous ions at the end Point.

11) **Why potassium ferrocyanide cannot be used as an indicator in estimation of iron ?**

Because it does not react with ferrous ions.

12) **What is reaction that occur during the titration?**

$K_2Cr_2O_7$ in acid medium oxidizes the ferrous ions in haematite ore solution to ferric Iron and gets itself reduced to chromic ions .

13) **What is the oxidation state of iron in haematite ore?**

Ferric, Fe^{3+} .

Demonstration Experiments

Experiment No.1

Flame photometric estimation of sodium and potassium.

1) **What is the basic principle of Flame photometry?**

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

2) **Name the filter used in the estimation of sodium/potassium by Flame photometry.**

sodium/potassium filter.

3) **What is the wavelength used in the estimation of sodium/potassium by Flame photometry?**

598nm/ 766 nm

4) **How do you estimate the sodium/potassium content in the given sample by Flame photometry?**

By plotting a calibration curve of ionization potential against concentration of sodium/potassium solution. From the calibration curve compute the concentration of sodium/potassium in the given sample.

5) **How do you express the concentration of sodium/Potassium in the water sample by Flame photometry?**

It is expressed in terms of mg of sodium/potassium.

6) **Give the sequence of events that occur in flame photometry**.

Liquid sample containing element aspirated into a flame → Formation of the liquid droplets → Evaporation of droplets resulting in the formation of residue → Decompose of residue into neutral atoms → Formation of excited atoms and emission of radiation from atoms and emission of radiation from atoms → wavelength and intensity of emitted radiation measured by flame photometry.

Experiment No.2

Synthesis of ZnO nano particle by Precipitation method

1) what is nano material?

A material having one of its dimension in the range of 1-100nm is called as nano material

2) what are the approaches to the synthesis of nanomaterials?

There are two approaches to the synthesis of nanomaterials

1) Bottom-up approach
2) Top-down approach

3) what are the advantages of synthesis of nanomaterials by precipitation method?

1) Simple & rapid method of preparation of nano materials.
2) Particle size & crystallinity can be controlled by optimizing conditions.

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