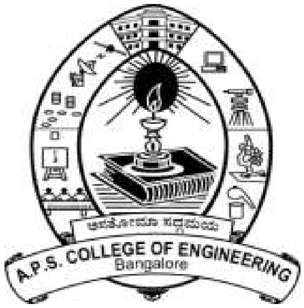


APPLIED CHEMISTRY LAB MANUAL

APS COLLEGE OF ENGINEERING

Anantha Gnana Gangothri Campus, Somanahalli,
Kanakapura Road, Bengaluru - 5600116

Vision:

To educate, train and inspire all students to become competent technocrats and help them emerge as leaders in every field of Engineering and to fill their hearts and minds with values of excellence in all their professional pursuits.

Mission:

1. To provide an excellent infrastructure and conducive ambience in the Institute.
2. To impart quality technical education combined with ethical values.
3. To inculcate attitude for Research and Development activities.
4. To establish platforms to connect with industries, educational institute of eminence and governmental organizations.

Department of Applied Sciences

Vision:

Department strives to be recognized for laying a strong foundation in basic sciences and prepare students to learn engineering better.

Mission:

1. To provide a platform to keep abreast with current development in science.
2. Acquire abilities to evaluate problems using Mathematics.
3. To create human resources with strong foundation in Physics, which can be applied to wide areas in Science and Technology.
4. To awaken the young minds and discover their talents both in theory and practical Chemistry.

APS COLLEGE OF ENGINEERING

Anantha Gnana Gangothri Campus, Somanahalli,
Kanakapura Road, Bengaluru - 5600116

PROGRAM OUTCOMES (PO's)

1. **Engineering knowledge:** Apply the knowledge of mathematics, science, engineering fundamentals, and an engineering specialization to the solution of complex engineering problems.
2. **Problem analysis:** Identify, formulate, review research literature, and analyze complex engineering problems reaching substantiated conclusions using first principles of mathematics, natural sciences, and engineering sciences.
3. **Design/development of solutions:** Design solutions for complex engineering problems and design system components or processes that meet the specified needs with appropriate consideration for the public health and safety, and the cultural, societal, and environmental considerations.
4. **Conduct investigations of complex problems:** Use research-based knowledge and research methods including design of experiments, analysis and interpretation of data, and synthesis of the information to provide valid conclusions.
5. **Modern tool usage:** Create, select, and apply appropriate techniques, resources, and modern engineering and IT tools including prediction and modeling to complex engineering activities with an understanding of the limitations.
6. **The engineer and society:** Apply reasoning informed by the contextual knowledge to assess societal, health, safety, legal and cultural issues and the consequent responsibilities relevant to the professional engineering practice.
7. **Environment and sustainability:** Understand the impact of the professional engineering solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.
8. **Ethics:** Apply ethical principles and commit to professional ethics and responsibilities and norms of the engineering practice.
9. **Individual and team work:** Function effectively as an individual, and as a member or leader in diverse teams, and in multidisciplinary settings.
10. **Communication:** Communicate effectively on complex engineering activities with the engineering community and with society at large, such as, being able to comprehend and write effective reports and design documentation, make effective presentations, and give and receive clear instructions.
11. **Project management and finance:** Demonstrate knowledge and understanding of the engineering and management principles and apply these to one's own work, as a member and leader in a team, to manage projects and in multidisciplinary environments.
12. **Life-long learning:** Recognize the need for, and have the preparation and ability to engage.

Applied Chemistry Laboratory

DO’S

- **Be punctual, follow the prescribed dress code.**
- **Follow all safety precautions displayed in the lab.**
- **Before using any chemicals, read the labels carefully.**
- **Report any accidents, injuries and breakage of glassware immediately to the concerned.**
- **Before leaving the laboratory, make sure that your work area is clean.**

DONT’S

- **Do not walk barefooted in the Chemistry lab.**
- **Do not touch chemicals or lab equipments unless you are asked to do so.**
- **Never taste any chemical.**
- **Eating, drinking, using mobile phones is strictly prohibited in the lab.**
- **Do not pour water into concentrated acids.**

INSTRUMENTATION EXPERIMENTS

EXPERIMENT 1

ESTIMATION OF FAS POTENTIOMETRICALLY USING STANDARD $K_2Cr_2O_7$ SOLUTION

Principle:

The procedure of using a measurement of e.m.f. to determine the concentration of ionic species in solution is referred to as potentiometry. The principle involved in potentiometric titration is the measurement of e.m.f. between two electrodes; an indicator electrode, the potential of which is a function of the concentration of the ion to be determined and a reference electrode (calomel electrode) of constant potential. In these titrations, the measurements of e.m.f. are made while the titration is in progress. The equivalence point of the reaction is revealed by a sudden change in potential in the plot of e.m.f. readings against the volume of titrant.

The determining factor in oxidation-reduction reactions is the ratio of the concentration of the oxidised and reduced forms of certain ion species. For the reaction:

$Cr^{6+} + 3e^- \rightarrow Cr^{3+}$, the potential, E acquired by the indicator electrode at 25^0C is given by

$$E = E^0 + \frac{0.0591}{n} \log_{10} \frac{[Cr^{6+}]}{[Cr^{3+}]} \qquad n = 3$$

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

Procedure :

Pipette out 50 cm^3 of ferrous ammonium sulphate solution into a 250 cm^3 beaker. Add two test tube of dilute sulphuric acid (2.5 M). Place the electrode assembly (platinum electrode as indicator electrode and a SCE as reference electrode) in the beaker and connect the same to the pH / Potentiometer. Read the potential difference between the electrodes with the aid of the meter (by using the meter in millivolt mode) and record it. Add 0.5 cm^3 of $K_2Cr_2O_7$ solution from a burette, mix the contents and after waiting for a half-minute, measure the e.m.f. of the cell. Repeat the addition of 0.5 cm^3 portions of $K_2Cr_2O_7$,. mixing and measuring the e.m.f. after each addition until a sudden change in the e.m.f. is observed. Henceforth, add the solution in portions of 0.5 cm^3 and record the readings after each addtion. Take 6 more readings.

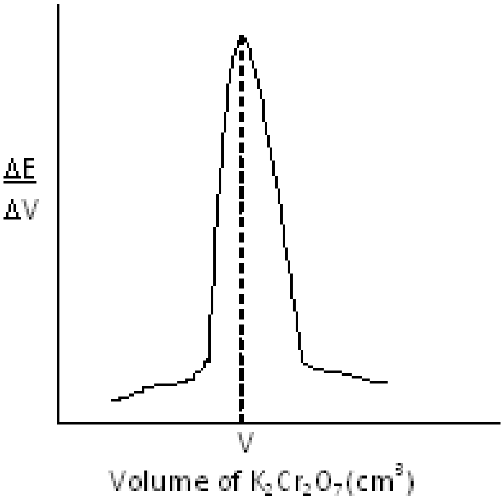
Plot e.m.f. (ordinate) versus volume of $K_2Cr_2O_7$ added (abscissa). Draw a smooth curve through the points. The equivalence point is the volume corresponding to the steepest portion of the curve. Locate the equivalence point of titration by plotting $\Delta E/\Delta V$ (ordinate) versus volume of titrant, (abscissa). There is a maximum in the plot at the equivalence point.

Result: Amount of FAS present in 50 cm^3 of the given solution =

OBSERVATION AND CALCULATION

ESTIMATION OF FAS POTENTIOMETRICALLY USING STANDARD $K_2Cr_2O_7$ SOLUTION.

Volume of $K_2Cr_2O_7$ added (cm^3)	**EMF (E) mV**	**ΔV**	**ΔE**	**$\frac{\Delta E}{\Delta V}$**
0.0		—	—	—
0.5				
1.0				



CALCULATION

Normality of $K_2Cr_2O_7$ = (To be given)

Volume of $K_2Cr_2O_7$ added = 'V' cm^3 (Equivalence point from graph)

Volume of FAS used = 50 cm^3

$$\therefore \text{, Normality of FAS} = \frac{(N \times V) \text{ of } K_2Cr_2O_7}{\text{Vol. of FAS}}$$

$\therefore$, Weight of FAS per litre = Normality x Equivalent wt. of FAS (392)

= .... g

$$\therefore \text{, Weight of FAS/50 } cm^3 = \frac{\text{................} \times 50}{1000} = \text{.......................................} \text{ g}$$

EXPERIMENT 2

CONDUCTOMETRIC ESTIMATION OF AN ACID MIXTURE USING STANDARD NaOH SOLUTION

Theory:

Measurement of conductance can be employed to determine the end point in acid-base titrations. In conductometric titrations, there is a sudden change in conductance of the solution at the equivalence point. The principle underlying conductometric titrations is the substitution of ions of one mobility by ions of another mobility. Therefore, the conductance of a solution depends on the number and mobility of ions. The equivalence point is determined graphically by plotting conductance (ordinate) against titre values (obscissa).

Conductometric titration may be applied for the determination of acids present in a mixture. In the titration of a mixture of a strong acid (HCl) and a weak acid (CH_3COOH) with a strong base (NaOH), the conductance initially decreases upon adding NaOH to acid mixture owing to the substitution of highly mobile H^+ ion (mobility: 350 $ohm^{-1}m^{-1}$) by the less mobile Na^+ ion (mobility: 50$ohm^{-1}m^{-1}$). This trend continues till all the H^+ ions of HCl replaced i.e., the strong acid is neutralised. Continued addition of NaOH rises the conductance moderately, as the weak acid CH_3COOH is converted into its salt, CH_3COONa. Further addition of NaOH rises the conductance steeply due to the presence of free OH^- ions (mobility: 198 ohm^{-1} m^{-1}). The titration curves in the graph given depict the location of the equivalence point.

Estimation of HCl

Pipette out 50 cm^3 of the given acid mixture into a clean 100 cm^3 beaker. Dip the conductivity cell in the solution and note down the conductance of the solution i.e., when the volume of NaOH added is zero.

Now add standard NaOH solution from a burette in increments of 0.5 cm^3. After each addition, stir the solution gently and note down the conductance. As the titration proceeds, the conductance first gradually decreases and then rises slowly and finally rises sharply. Continue titration until the conductance is more or less the same as it was in the beginning. Plot a graph of conductance on Y- axis versus volume of NaOH on X-axis to get three straight lines as shown in the figure. The point 'V_1' gives the volume of sodium hydroxide needed to neutralize hydrochloric acid and the point 'V_2' gives the volume of NaOH needed to neutralize both HCl and CH_3COOH. The difference in the volumes i.e., ($V_2 - V_1$) gives the volume of NaOH needed to neutralize CH_3COOH.

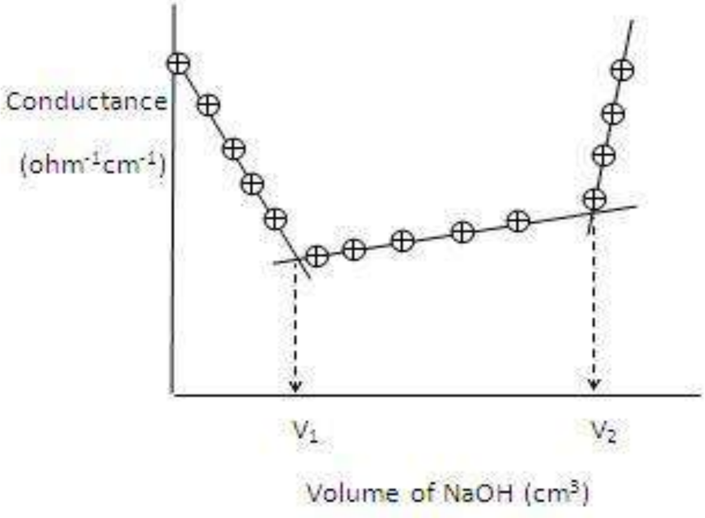
Result: Amount of HCl/dm^3 of the solution =

Amount of CH_3COOH/dm^3 of the solution =

OBSERVATION AND CALCULATION

CONDUCTOMETRIC ESTIMATION OF HCl and CH_3COOH USING STANDARD NaOH SOLUTION

Vol. of NaOH (cm^3)	Conductance $(ohm^{-1}\ cm^{-1})$	Vol. of NaOH (cm^3)	Conductance $(ohm^{-1}\ cm^{-1})$
0.0		6.5	
0.5		7.0	
1.0		7.5	
1.5		8.0	
2.0		8.5	
2.5		9.0	
3.0		9.5	
3.5		10.0	
4.0		10.5	
4.5		11.0	
5.0		11.5	
5.5		12.0	
6.0		12.5	



Estimation of HCl

Volume of the acid mixture taken = 50 cm^3

Normality of NaOH = 1.0

Volume of NaOH required to neutralise HCl = V_1 cm^3

$$\text{Normality of HCl} = \frac{\text{Normality of NaOH x Volume of NaOH } (V_1)}{50}$$

Therefore, the weight of HCl/dm^3 = Normality of HCl x Eq.wt. of HCl (36.5)

Estimation of CH_3COOH

Volume of NaOH required to neutralise CH_3COOH = $(V_2 – V_1)$ cm^3

$$\text{Normality of } CH_3COOH = \frac{\text{Normality of NaOH x Volume of NaOH } (V_2 - V_1)}{50}$$

Therefore, the weight of CH_3COOH /dm^3 = Normality of CH_3COOH x Eq.wt. of CH_3COOH (60)

EXPERIMENT 3

DETERMINATION OF VISCOSITY COEFFICIENT OF A LUBRICANT USING OSTWALD'S VISCOMETER

Principle:

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 drag or friction on its nearest moving layer backwards. This property of a liquid by which it retards or opposes motion between layers is called viscosity. The coefficient of viscosity is defined as the tangential force per unit area required to maintain a unit velocity gradient between any two successive layers of a liquid situated unit distance apart. The coefficient of viscosity of a liquid is given by Poiseuille's formula

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

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 coefficient of viscosity of the liquid. If equal volumes of two different liquids are allowed to flow through the same tube under identical conditions, then,

$$\frac{\eta_l}{\eta_w} = \frac{t_l \, d_l}{t_w \, d_w}$$

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

Procedure:

Clean the given viscometer with acetone very well and dry it in a hot air oven and remove the vapours by using suction pump. Fix the viscometer vertically to a stand. Using a pipette, transfer a known volume (say 20 cm^3) of **distilled water only into the wider limb**. Suck it above the upper mark of the viscometer. Allow it to flow freely through the capillary. When the level of water just crosses the upper mark (A) start a stop clock. Stop the stop clock when the water just crosses the lower mark (B). Record the time of flow in seconds (t_w). Repeat for 2 times.

Pour out the water, rinse the viscometer and pipette with acetone and dry as before. Clamp the viscometer vertically to a stand and transfer the same volume (20 cm^3) of the test liquid (lubricant) into the wider limb. As described above, record the time (t_l) taken in seconds by the lubricant to flow through the same distance. Determine the coefficient of viscosity of the lubricant using the relation,

$$\frac{\eta_l}{\eta_w} = \frac{t_l \; d_l}{t_w \; d_w} \qquad \eta_l = \frac{t_l \; d_l \; x \; \eta_w}{t_w \; d_w}$$

Result: Viscosity coefficient of the given lubricant =

OBSERVATION AND CALCULATION

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

	FLOW TIMES	
Water	Time of flow (seconds)	
	1.	
	2.	
	3.	
	Mean time, t_w	
Given liquid	1.	
	2.	
	3.	
	Mean time, t_l	

CALCULATION

Laboratory temperature = ^{0}C

Density of water at ^{0}C = ------------------- g/cm^3

Viscosity coefficient of water at ^{0}C = ------------------- millipoise

Density of the given lubricant at ^{0}C = ---------------- g/cm^3

Viscosity coefficient of the given lubricant, $\eta_l = \frac{d_l \, x \, t_l}{d_w \, x \, t_w} \; x \; \eta_w$ = --------------- millipoise

RESULT : The viscosity coefficient of the given lubricant =-------------------- millipoise

EXPERIMENT 4

ESTIMATION OF COPPER PRESENT IN ELECTROPLATING EFFLUENT BY OPTICAL SENSOR (COLORIMETRY)

Principle:

When a monochromatic light of intensity I_0 is incident on a transparent medium, a part, I_a of it is absorbed, a part, I_r is reflected 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 and therefore,

$$I_0 = I_a + I_t$$

$I_t / I_0 = T$ called the transmittance, $\log 1/T = \log I_0 / I_t$ is called the absorbance or optical density. The relation between absorbance, A, concentration, c (expressed in mol/dm^3) and path length, t (expressed in cm) is given by **Beer-Lambert's law**,

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

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

A series of standard solutions of copper sulphate penta hydrate is treated with ammonia to get blue cuprammonium complex, $[Cu(NH_3)_4]^{2+}$ and is diluted to a definite volume. 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.

The given 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: (Note: use distilled water only)

Transfer the given copper sulphate solution (stock solution) to a burette and draw out 2, 4, 6, 8 and 10 cm^3 of the solution into 50 cm^3 volumetric flasks. Now collect the test solution from the teacher / examiner, add 5 cm^3 of ammonia solution to each of them, shake well and then dilute up to the mark with ion exchange water. Stopper the flasks and mix the solutions well. To the **test solution (electroplating effluent)** given in a 50 cm^3 measuring flask, also add 5 cm^3 of ammonia solution, shake well and then dilute up to the mark with **distilled water and mix well.** Prepare a blank solution by diluting only 5 cm^3 of ammonia solution in a 50 cm^3 measuring flask up to the mark with distilled water and mixing well. After 10 minutes, measure the absorbance of the solutions against blank at **620 nm** using a photoelectric colorimeter. Tabulate the readings as shown. Draw a calibration curve by plotting absorbance against volume of copper sulphate solution. Using the calibration curve, find out the volume of copper sulphate solution given i.e., the volume of test solution and calculate the amount of copper in the given effluent solution.

Result: The amount of copper present in the given electroplating effluent =

OBSERVATION AND CALCULATION

ESTIMATION OF COPPER PRESENT IN ELECTROPLATING EFFLUENT BY OPTICAL SENSOR (COLORIMETRY)

Volume of copper sulphate solution (cm^3)	Concentration of copper = 2.037 mg x vol. of solution	Absorbance (optical density)
Blank solution	-----	
2.0		
4.0		
6.0		
8.0		
10.0		
Test solution		

CALCULATION

1000 cm^3 of stock solution contains 8 g of $CuSO_4 . 5H_2O$

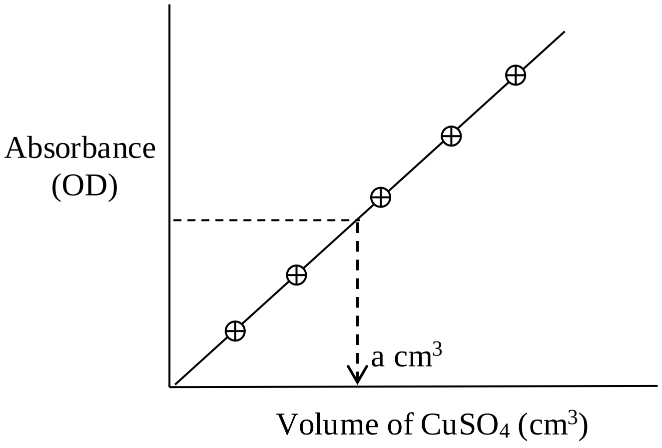
249.54 g of $CuSO_4.5H_2O \equiv$ 63.54 g of Cu

8 g of $CuSO_4.5H_2O \equiv$ 63.54 x 8 / 249.54 = 2.037 g of Cu per 1000 cm^3 of stock solution

$\therefore$, 1 cm^3 of $CuSO_4.5H_2O \equiv$ 2.037/1000 = 0.002037 g of Cu = 2.037 mg of Cu

$\therefore$, Cu present in 'a' cm^3 of test solution = 'a' cm^3 x 2.037 mg = ... mg

RESULT: The weight of copper in the given test solution =.......................... mg



EXPERIMENT 5
DETERMINATION OF pK_a VALUE OF VINEGAR USING pH SENSOR

Principle:

The strength of an acid is experimentally measured by determining its equilibrium constant or dissociation constant (K_a). Since strong acids are strong electrolytes, they are ionised almost completely in aqueous solutions. It is not meaningful to study the ionic equilibrium of strong acids and calculate their equilibrium constants as the unionised form is present to such a small extent. Hence, the study of ionic equilibrium and calculation of K is applicable only to weak acids. For eg.Acetic acid (vinegar) ionises feebly as, $CH_3COOH(aq) + H_2O(l) \longrightarrow H_3O^+(aq) + CH_3COO^-(aq)$

$$K_a = \frac{[H_3O^+][CH_3COO^-]}{[CH_3COOH]}$$

pK_a is a modern method of expressing acid strengths. $pK_a = -\log_{10} K_a$

pK_a is determined by measuring the changes in pH of acid solution at different amounts of the base added.

During the titration of an acid with a base, the pH of the solution rises gradually at first, then more rapidly and until at the equivalence point, there is a very sharp increase in pH for a very small quantity of added base. Once past the equivalence point, the pH increases only slightly on addition of excess base. The titration curve is obtained by plotting changes in pH at different amounts of the base added and the equivalence point is determined.

According to Henderson-Hasselbalch equation, $pH = pK_a + \log_{10} \frac{[Salt]}{[Acid]}$

At half equivalence point, [salt] = [acid] and therefore, pH at half equivalence point gives the pK_a of weak acid.

Procedure:

Pipette out 50 cm^3 of the given vinegar into a 100 cm^3 beaker. Immerse a glass electrode-calomel electrode assembly into the acid and connect the cell to a pH meter. Measure the pH of the acid. Fill a burette with the base (sodium hydroxide). In the beginning, add large increments of (say 0.5 cm^3) of the base to the acid. Stir the solution thoroughly and measure the pH after each addition. When the pH begins to show a tendency to increase rapidly, add only small increments (say 0.5 cm^3) of the base and measure the pH after each addition. Continue till there is only a slight increase in pH on the addition of the base.

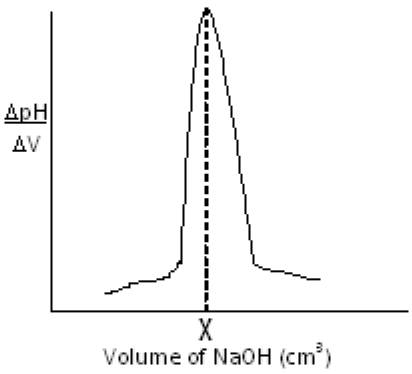
Plot a graph of pH (ordinate) against the volume of sodium hydroxide added (abscissa). Determine the equivalence point and hence the pH at half equivalence point. This gives the pK_a value of the acid.

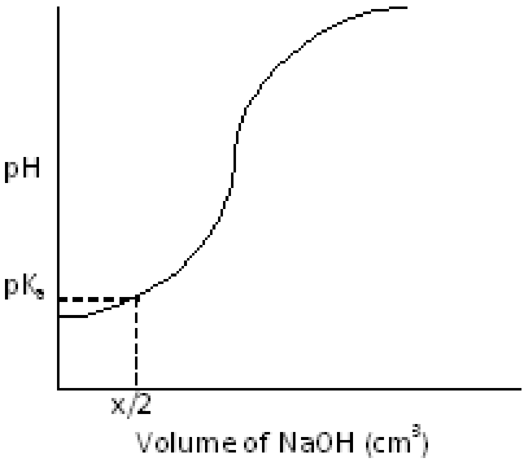
Result: pKa value of the given vinegar solution =

OBSERVATION AND CALCULATION

DETERMINATION OF pK_a VALUE OF VINEGAR (WEAK ACID) USING pH SENSOR

Volume of NaOH added (cm^3)	pH	ΔV	ΔpH	$\frac{\Delta pH}{\Delta V}$
0.0		--------	--------	
0.5		0.5		
1.0		0.5		
1.5		0.5		
2.0		0.5		
2.5		0.5		
3.0		0.5		
3.5		0.5		
4.0		0.5		
4.5		0.5		
5.0		0.5		
5.5		0.5		
6.0		0.5		
6.5		0.5		
7.0		0.5		





1) Equivalence point = x = ...

2) Half equivalence point = $x/2$ = ...

3) pH at half equivalence point = pK_a = ...

4) pK_a of the given weak acid(vinegar) = pH at half equivalence point = ...

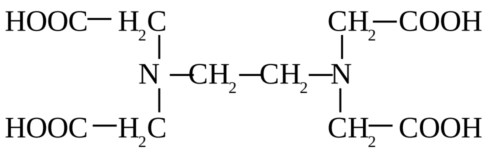
VOLUMETRIC EXPERIMENTS

EXPERIMENT 1

ESTIMATION OF TOTAL HARDNESS OF WATER BY EDTA METHOD

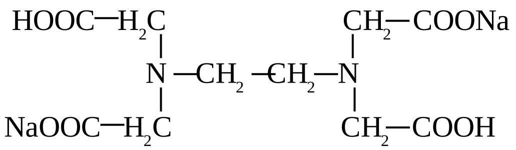
Principle:

Hardness of water is due to the presence of calcium and magnesium salts in water. Ethylenediaminetetraacetic acid (EDTA) forms complexes with a large number of cations including Ca^{2+} and Mg^{2+} ions. Accordingly, it is possible to determine the total hardness of water using EDTA reagent.



$$\begin{array}{ccc} \mathrm{HOOC-H_2C} & & \mathrm{CH_2-COOH} \\ & \mathrm{N-CH_2-CH_2-N} & \\ \mathrm{HOOC-H_2C} & & \mathrm{CH_2-COOH} \end{array}$$

EDTA (H_4Y)



$$\begin{array}{ccc} \mathrm{HOOC-H_2C} & & \mathrm{CH_2-COONa} \\ & \mathrm{N-CH_2-CH_2-N} & \\ \mathrm{NaOOC-H_2C} & & \mathrm{CH_2-COOH} \end{array}$$

Na_2H_2Y

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

$$M^{2+} + H_2Y^{2-} \rightarrow MY^{2-} + 2H^{+} \quad .... (1)$$

where M^{2+} is Ca^{2+} and Mg^{2+} present in water. Reaction (1) can be carried out quantitatively at a pH of 10 using Eriochrome Black T indicator. Since the reaction involves the liberation of H^+ ions, a buffer mixture has to be used to maintain a pH of 10. The buffer mixture used in the titration is NH_3-NH_4Cl. The hardness of water is usually expressed in terms of ppm (parts per million) of $CaCO_3$. Since EDTA (free acid) is sparingly soluble, its disodium salt, Na_2H_2Y is used for preparing the reagent.

Procedure:

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

Weigh the weighing bottle containing about 2.5 g of disodium salt of EDTA accurately using an electronic balance and note the weight. Transfer the crystals carefully onto a funnel placed over a 250 cm^3 volumetric flask and record the weight of the empty weighing bottle. Pour ion exchange water through the funnel allowing all the crystals to run down into the flask. Add 5 ml Ammonia solution. Wash the funnel with ion exchange water and remove the funnel. Dissolve the crystals by swirling the flask gently. Dilute the solution up to the mark with ion exchange water, stopper the flask and mix the solution thoroughly by inverting the flask several times so that a homogeneous solution results.

Part B: Determination of total hardness of water sample

Pipette out 25 cm^3 of the given water sample into a clean conical flask. Add 3 cm^3 of NH_3-NH_4Cl buffer and a pinch/few drop of Eriochrome Black T indicator. Titrate against standard EDTA solution till the colour of the solution changes from wine red to clear blue. Perform the titration slowly near the end point.

Result: The total hardness of given water sample =

OBSERVATION AND CALCULATION

DETERMINATION OF TOTAL HARDNESS OF A SAMPLE OF WATER

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

1. Weight of bottle + EDTA salt = ... g
2. Weight of empty bottle = ... g
3. Weight of EDTA salt = ... g

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

Part B: Determination of total hardness of water sample

Burette : Standard EDTA solution

Conical flask : 25 cm^3 water sample + 3 cm^3 of NH_3-NH_4Cl buffer

Indicator : Eriochrome Black T

End point : Wine red to clear blue

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

CALCULATION

Volume of EDTA used = ... cm^3

1000 cm^3 of 1M EDTA ≡ 100 g of $CaCO_3$ (molecular mass of $CaCO_3$ = 100)

$$\text{................ } cm^3 \text{ of ....M EDTA} \equiv \frac{\text{........... x .... x 100}}{1000 \times 1}$$

= ....c g of $CaCO_3$ / 25 cm^3 of hard water

$$\therefore\text{, Total hardness of the given water sample in parts per million (ppm)} = \frac{c \times 10^6}{25} \text{ ppm of } CaCO_3$$

= ---------- ppm of $CaCO_3$

EXPERIMENT 2

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

Principle:

The chemical oxygen demand (COD) test is extensively employed as a means of measuring the pollutional strength of industrial wastes. Chemical oxygen demand is a measure of the total quantity of oxygen required for complete oxidation of organic compounds of wastes to CO_2 and water by a strong oxidising agent. This parameter is particularly valuable in surveys designed to determine the losses to sewer systems. Results may be obtained within a relatively short time and measures taken to correct errors on the day they occur.

Waste water contains organic impurities which include straight chain aliphatic compounds, aromatic hydrocarbons, straight chain alcohols, acids and other oxidisable materials. Straight chain compounds, acetic acid, etc., are oxidised more effectively when silver sulphate is added as a catalyst. But silver sulphate reacts with chlorides in the waste water to form precipitates which are oxidised only partially by this procedure. This difficulty is overcome by adding mercuric sulphate to the sample.

A known volume of the waste water is refluxed with excess of potassium dichromate solution in sulphuric acid medium and in the presence of Ag_2SO_4 and $HgSO_4$. Potassium dichromate oxidizes all oxidisable impurities. The unreacted dichromate is estimated by titrating against standard FAS solution (Back titration) using ferroin indicator. The amount of $K_2Cr_2O_7$ consumed corresponds to the COD of waste water sample. To calculate this, a blank titration without waste water sample is carried out.

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

$$\underset{\text{Bluish green}}{Fe(C_{12}H_8N_2)_3^{3+}} + e \rightarrow \underset{\text{Reddish brown}}{Fe(C_{12}H_8N_2)_3^{2+}}$$

(1,10-phenanthroline-iron II complex) Ferroin

Procedure:

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

Weigh about 2.5 grams of Mohr’s salt accurately into a 250 cm^3 volumetric flask. **Add two test tubes of dilute sulphuric acid** and dissolve the crystals. Dilute the solution with ion exchange water up to the mark and shake well.

Back Titration :

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

Blank Titration :

Pipette out 10 cm^3 of standard potassium dichromate solution. Add 1 test tube of 6N sulphuric acid (containing silver sulphate), reflux for half an hour. Cool and add 2-3 drops of ferroin indicator. Titrate against standard Mohr's salt solution until the colour turns from blue green to reddish brown.

***Note: In practice, boiling and cooling need not be done, carryout as such.**

Result: COD of the given industrial waste water sample =

OBSERVATION AND CALCULATION
DETERMINATION OF CHEMICAL OXYGEN DEMAND (COD)

Part A: Preparation of standard Ferrous Ammonium Sulphate (FAS) solution (Mohr's salt)

1. Weight of bottle + FAS crystals = ... g
2. Weight of empty bottle = ... g
3. Weight of FAS crystals = ... g

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

Back Titration-Estimation of COD of waste water sample

Burette : Standard FAS solution

Conical flask : 25 cm^3 of waste water sample + 10 cm^3 of standard potassium dichromate solution + 2 t.t. of 6N H_2SO_4 + boiling chips, reflux the mixture for half an hour, cool

Indicator : 2-3 drops of ferroin

End point : Blue green to reddish brown

Burette Reading	Trail-I	Trail-II	Trail-III
Final Reading			
Initial Reading			
Volume of FAS run down in cm^3			

Volume of FAS used for back titration = ... cm^3 ---b

(i.e. the volume of FAS consumed by unreacted $K_2Cr_2O_7$)

Blank Titration (value to be given by the examiner)

Volume of FAS used for blank titration = ... cm^3 ---c

CALCULATION

1000 cm^3 of 1N FAS solution ≡ 1 equivalent of oxygen ≡ 8 g of oxygen (or 8 x 10^3 mg of O_2)

$$\text{(c-b) cm}^3 \text{ of ......N FAS solution} = \frac{8 \times 10^3 \times (c-b) \times \text{N of FAS}}{1000 \times 1} = \text{.......... mg of oxygen/ 25cm}^3 \text{ of waste water}$$

$$\therefore, \text{1000 cm}^3 \text{ of waste water sample contains} = \frac{\text{................... x 1000}}{25} \text{ mg of oxygen / dm}^3$$

$\therefore$, COD of waste water sample = ... mg of O_2 per dm^3

EXPERIMENT 3

ESTIMATION OF PERCENTAGE OF IRON IN TMT BAR USING STANDARD POTASSIUM DICHROMATE SOLUTION (EXTERNAL INDICATOR METHOD)

Principle:

Thermo mechanically tested bars (TMT bars) are essential for all type of construction purposes. It is manufactured using virgin iron ore. TMT bar is electrolysed in a solution of sodium bicarbonate (an inert electrolyte) to form ferric oxide. Ferric oxide so formed is treated with 1:1 HCl to form $FeCl_3$. Since iron in $FeCl_3$ is already in the highest possible oxidation state (Fe^{3+}), it is first reduced to ferrous state by using a slight excess of stannous chloride. The excess of stannous chloride added is destroyed by adding saturated solution of mercuric chloride, since $SnCl_2$ can reduce potassium dichromate. Then, the solution is titrated with standard solution of $K_2Cr_2O_7$ using potassium ferricyanide as an external indicator. The volume of $K_2Cr_2O_7$ consumed is a measure of the amount of iron present in rust solution.

$$\underset{\text{Ferric chloride}}{2FeCl_3} + \underset{\text{Stannous chloride}}{SnCl_2} \rightarrow \underset{\text{Ferrous chloride}}{2FeCl_2} + \underset{\text{Stannic chloride}}{SnCl_4}$$

$$SnCl_2 + \underset{\text{Mercuric chloride}}{2HgCl_2} \rightarrow \underset{\text{Mercurous chloride}}{Hg_2Cl_2\downarrow} + SnCl_4$$

$$(Fe^{2+} \rightarrow Fe^{3+} + e^-) \times 6 \qquad ....(1)$$

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

Adding (1) & (2)

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

$$2K_3[Fe(CN)_6] + 3Fe^{2+} \rightarrow \underset{\text{Ferrous ferricyanide (Blue)}}{Fe_3[Fe(CN)_6]_2} + 6K^+$$

Procedure :

Part A: Preparation of standard $K_2Cr_2O_7$ solution

Weigh about 1.25 g of potassium dichromate crystals accurately and transfer to a 250 cm^3 volumetric flask. Dissolve in ion exchange water and dilute up to the mark and mix well.

Part B: Estimation of Iron

Pipette out 25 cm^3 of the TMT bar solution into a clean conical flask. Add a quarter test tube of concentrated HCl and heat the solution to boiling. Add stannous chloride to the hot solution drop wise till the yellow solution turns colourless. Add 2 more drops to ensure complete reduction. Cool and add a quarter test tube of mercuric chloride rapidly. A silky white precipitate of mercurous chloride is formed. Add one test tube of ion exchange water. Place a number of drops of freshly prepared potassium ferricyanide indicator on a wax paper. Add a small quantity of potassium dichromate from a burette to the conical flask containing rust solution and mix well. Remove a drop of the solution from the conical flask and bring it in contact with a drop of the indicator on the wax paper. The colour of the indicator turns blue. Repeat this operation after adding 1 cm^3 more of the potassium dichromate solution. The indicator turns blue as long as the titration is incomplete. Continue the titration by adding increments of 1 cm^3 of $K_2Cr_2O_7$ at a time and testing as above till a drop of the mixture fails to produce any colour with the indicator drop. ***(Note: Clean the glass rod after every test).*** Repeat the titration by taking another 25 cm^3 of the TMT bar solution. This time add most of the potassium dichromate solution required at a stretch and then titrate dropwise. Mix the contents of the flask after every addition and test a drop of the titrated mixture with a drop of the indicator as described above till the colour of the indicator drop does not change. Repeat for agreeing values.

Result: The percentage of iron present in the given TMT bar solution =

OBSERVATION AND CALCULATION

ESTIMATION OF PERCENTAGE OF IRON IN THE GIVEN TMT BAR SOLUTION

Part A: Preparation of standard $K_2Cr_2O_7$ solution

1. Weight of bottle + $K_2Cr_2O_7$ crystals = ... g

2. Weight of empty bottle = ... g

3. Weight of $K_2Cr_2O_7$ crystals = ... g

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

Part B: Estimation of Iron

Burette : Standard $K_2Cr_2O_7$ solution

Conical flask : 25 cm^3 of TMT bar solution + ¼**th** t.t con. HCl, heat + $SnCl_2$ dropwise till colourless + 2 drops in excess, cool + ¼**th** t.t $HgCl_2$, shake well + 1 t.t. H_2O

Indicator : [K_3 (Fe(CN)$_6$] (external)

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

Burette Reading	Trail-I	Trail-II	Trail-III
Final Reading			
Initial Reading			
Volume of $K_2Cr_2O_7$ run down in cm^3			

CALCULATION

Volume of $K_2Cr_2O_7$ used = ... cm^3

Weight of TMT bar in 25 cm^3 = ………. g (To be given)

1000 cm^3 of 1N $K_2Cr_2O_7$ ≡ 55.85 g of Fe (1 equivalent of Fe)

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

= .... g of Fe / 25 cm^3 of solution

$$\therefore, \text{ Percentage of iron in the given TMT bar solution} = \frac{\text{....................g} \times 100}{\text{Weight of TMT bar}}$$

EXPERIMENT A1
SYNTHESIS OF IRON OXIDE NANOPARTICLES

In recent years, iron oxide nanoparticles have demonstrated great potential in biomedical applications due to their non-toxic role in biological systems. Also, the magnetic and semiconductor properties of iron oxide nanoparticles can lead to multifunctional applications in medicine. These nanoparticles have been developed as antibacterial, antifungal, and anticancer. For cancer treatment and diagnosis, iron oxide nanoparticles have been functionalized with drugs. However, many of those drugs have been related to adverse effects on health. On the other hand, phytochemicals from extracts of plants have been used as an alternative for the functionalization of these nanoparticles preventing negative effects. The main advantage of these nanoparticles is the high biodistribution in the organism compared with other drug delivery systems. The magnetism of iron oxide nanoparticles has been used in cancer treatment and diagnosis, for example, thermoablation, hyperthermia, and contrast media in magnetic resonance imaging.

Iron oxide nanoparticles are synthesized by precipitation method, which is a simple technique among chemical route. This method is given more priority due to its low cost and easy industrialization with improved reproducibility. This method offers low temperature growth and scale-up fabrication which yields colloidal solutions with wide range of particle districutions.

Principle: Co-precipitation is a very facile and convenient way to synthesize iron oxide nanoparticles (Fe_3O_4) from aqueous Fe^{2+} / Fe^{3+} salt solutions by the addition of a base at room temperature or at elevated temperature.

Chemicals required – $FeSO_4.2H_2O$ (0.2M), $FeCl_3.6H_2O$ (0.1M),
Ammonia solution (3M) and deionized water

Procedure: 0.2M ferrous sulphate solution and 0.1M ferric chloride solutions are mixed with 100ml deionized water in a beaker. Ammonia solution (3M) is added dropwise with constant stirring at 1000 rpm using a magnetic stirrer till pH 10. The black precipitate of iron oxide obtained is filtered and dried in hot-air over at 150 – 200^{o}C.

Reaction:

$$2\,FeSO_4 + FeCl_3 + 5\,NH_3 \rightarrow Fe_3O_4 + (NH_4)_2SO_4 + 3\,NH_4Cl$$

EXPERIMENT A2
ELECTROLYSIS OF WATER

Electrolysis of water, also known as electrochemical water splitting, is the process of using electricity to decompose water into oxygen and hydrogen gas by electrolysis. Hydrogen gas released in this way can be used as hydrogen fuel, or remixed with the oxygen to create oxyhydrogen gas, which is used in welding and other applications.

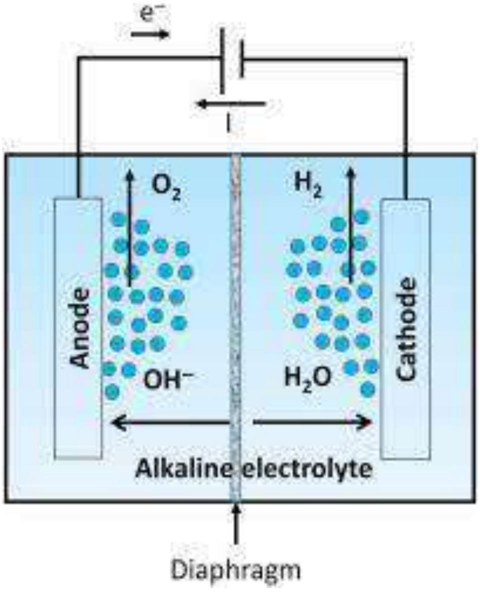
Electrolysis of water requires a minimum potential difference of 1.23 volts, though at that voltage external heat is required from the environment.

A DC electrical power source is connected to two electrodes, or two plates (typically made from an inert metal such as platinum or graphite) which are placed in the water. Hydrogen will appear at the cathode (where electrons enter the water), and oxygen will appear at the anode. Assuming ideal faradaic efficiency, the amount of hydrogen generated is twice the amount of oxygen, and both are proportional to the total electrical charge conducted by the solution. However, in many cells competing side reactions occur, resulting in different products and less than ideal faradaic efficiency.

Electrolysis of pure water requires excess energy in the form of over potential to overcome various activation barriers. Without the excess energy, the electrolysis of pure water occurs very slowly or not at all. This is in part due to the limited self-ionization of water. Pure water has an electrical conductivity about one-millionth that of seawater. Many electrolytic cells may also lack the requisite electrocatalysts. The efficiency of electrolysis is increased through the addition of an electrolyte (such as a salt, an acid or a base) and the use of electrocatalysts.

Alkaline electrolysis of water:

The important components used in the electrolyser are as shown in the figure.



Anode: Nickel metal particles dispersed on porous carbon is used as anode electrocatalyst.

Cathode: Nickel metal particles coated on porous carbon is used as cathode electrocatalyst.

Electrolyte: Aqueous solution of KOH (with 20 to 30 weight % KOH) is used as an electrolyte.

Separator: Porous dense anion exchange membrane is used as the separator. It is a good ionic conductor of hydroxyl ions and bad electronic conductor. It prevents the spontaneous recombination of H_2 and O_2.

Deionised water is passed in to the cathode chamber. At cathode water molecules are reduced to hydrogen and hydroxyl ions. The hydroxyl ions move through the separator towards anode and get oxidized to oxygen and water.

Reactions: At cathode: $2\ H_2O + 2\ e^- \rightarrow H_2(g) + 2\ OH^-$

At anode: $2\ OH^- \rightarrow \frac{1}{2}\ O_2(g) + H_2O + 2\ e^-$

Overall reaction: $H_2O \rightarrow H_2(g) + \frac{1}{2}\ O_2(g)$

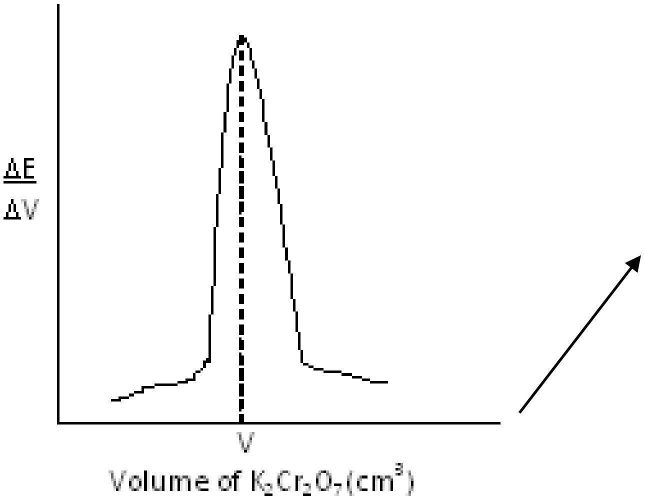
MODEL PROCEDURE WRITING

EXPERIMENT 1

ESTIMATION OF FAS POTENTIOMETRICALLY USING STANDARD $K_2Cr_2O_7$ SOLUTION

Pipette out 50 cm^3 of FAS solution into a beaker. Add 2 test tube dilute H_2SO_4. Immerse the calomel electrode - platinum electrode assembly into it. Connect the electrode assembly to a potentiometer and measure the potential. Add $K_2Cr_2O_7$ solution from a burette in increments of 0.5 cm^3 and measure the potential after each addition. Continue till there is sudden increase in potential and take few more readings.

Plot a graph of $\Delta E/\Delta V$ (ordinate) against volume of $K_2Cr_2O_7$ (abscissa) and determine the equivalence point.



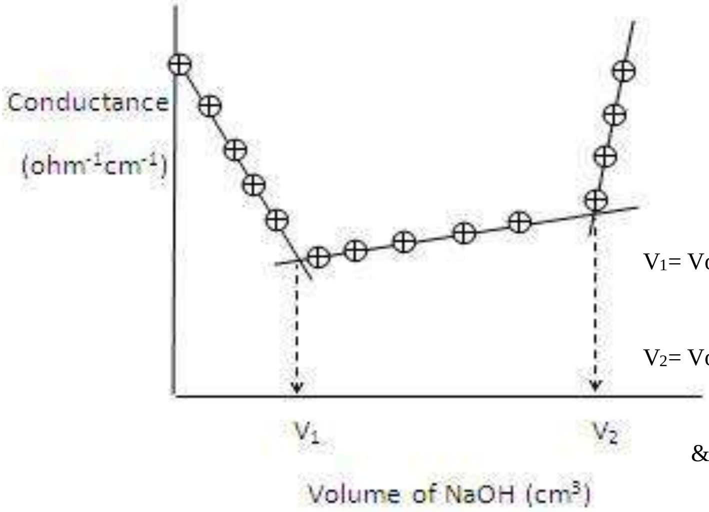
From the normality of $K_2Cr_2O_7$, calculate the normality and the weight of FAS in the given solution.

EXPERIMENT 2

CONDUCTOMETRIC ESTIMATION OF AN ACID MIXTURE USING STANDARD NaOH SOLUTION

Pipette out 50 cm^3 of given acid mixture into a beaker. Immerse the conductivity cell in it. Connect the cell to a conductivity meter and measure the conductance. Add 1N NaOH from the burette in increments of 0.5 cm^3 and measure the conductance after each addition. Continue titration until the conductance is more or less the same as it was in the beginning.

Plot the graph of conductance against (ordinate) volume of NaOH (abscissa) and determine the equivalence points. From the normality and volume of NaOH consumed, calculate the normality of HCl and CH_3COOH. Then calculate the amount of HCl and CH_3COOH in the given solution using the normality.



V_1= Vol. Of NaOH required neutralize HCl

V_2= Vol. Of NaOH required neutralize HCl & CH_3COOH

EXPERIMENT 3

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

Pipette out 20 cm^3 of distilled water into a wide limb of the clean and dry viscometer and suck the water through the other limb. Record the time of flow between two fixed points, one above and one below the bulb in the narrow limb of the viscometer. Repeat, record another two readings and take the average time of flow. Pour out the water, wash the viscometer with acetone and dry it. Now pipette out 20 cm^3 of the given lubricant into the wider limb and determine the average time of flow for liquid as before. Record the laboratory temperature. Calculate the viscosity coefficient of the given liquid (η_l) using the expression;

$$\eta_l = \frac{d_l \text{ X } t_l}{d_w \text{ X } t_w} \text{ X } \acute{\eta}_w$$

where, t_l = time of flow of lubricant

t_w = time of flow of water

d_l = density of lubricant

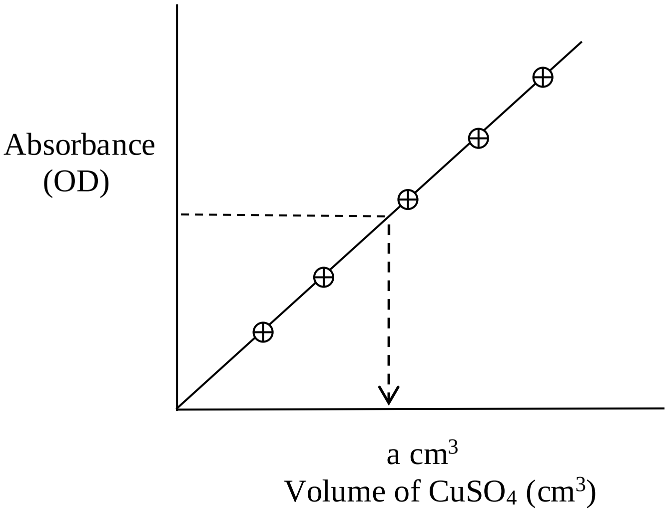
d_w = density of water

η_w = viscosity coefficient of water

EXPERIMENT 4

ESTIMATION OF COPPER PRESENT IN ELECTROPLATING EFFLUENT BY OPTICAL SENSOR (COLORIMETRY)

Draw out 2, 4, 6, 8, 10 cm^3 of given copper sulphate solution into five separate 50 cm^3 volumetric flasks. Add 5 cm^3 of NH_3 to each one of them and also into the test solution of unknown concentration, dilute up to the mark with distilled water and mix well. Measure the absorbance of each of these against blank solution (only ammonia and water) at 620 nm using a photoelectric colorimeter. Plot a graph of absorbance (OD) (ordinate) against concentration of copper (abscissa) and determine the concentration of copper in the test solution.

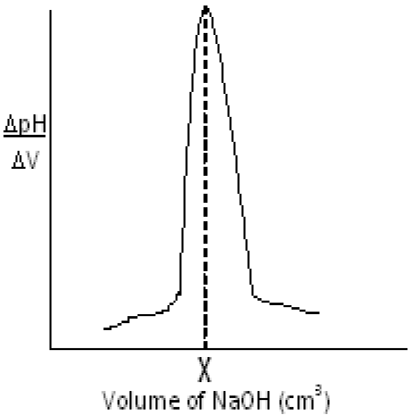


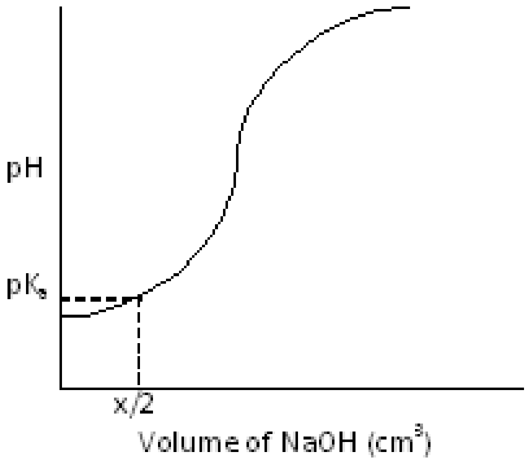
EXPERIMENT 5

DETERMINATION OF pK_a VALUE OF A WEAK ACID USING pH SENSOR

Pipette out 50 cm^3 of the given vinegar soltuion into a beaker. Immerse the glass electrode + calomel electrode assembly into it. Connect the electrodes to pH meter and measure the pH. Now add NaOH from a burette in increments of 0.5 cm^3 and measure the pH after each addition. Continue the titration until there is sudden increase in pH and take few more readings.

Plot a graph of $\Delta pH/\Delta V$ (ordinate) against volume of NaOH (abscissa) and determine the equivalence point. Plot another graph of pH (ordinate) against volume of NaOH (abscissa) and determine pK_a value of the given vinegar. pH at half equivalence point gives pK_a.





pH at $x/2 = pK_a$

EXPERIMENT 1

DETERMINATION OF TOTAL HARDNESS OF A SAMPLE OF WATER

Part A : Preparation of standard solution

Weigh out the given EDTA crystals accurately into a 250 cm^3 volumetric flask. Add 5 cm^3 of ammonia solution. Dissolve in distilled water and dilute up to the mark with distilled water. Mix well.

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

Part B : Estimation of total hardness

Burette : Standard EDTA solution
Conical flask : 25 cm^3 water sample + 3 cm^3 NH_3-NH_4Cl buffer solution (pH = 10)
Indicator : Eriochrome Black T
End point : Wine red to clear blue

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

EXPERIMENT 2

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

Part A : Preparation of standard solution

Weigh out the given ferrous ammonium sulphate (FAS) crystals accurately into a 250 cm^3 volumetric flask. Add 2 test tube dilute H_2SO_4, dissolve the crystals and dilute up to the mark with distilled water. Mix well.

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

Part B: Determination of COD of waster water sample

Burette : Standard FAS solution

Conical flask : 25 cm^3 waste water sample + 10 cm^3 $K_2Cr_2O_7$ (pipette out) + 2 test tube 6N H_2SO_4. Boil for half an hour, cool.

Indicator : Ferroin

End point : Blue green to reddish brown

Perform a 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.

EXPERIMENT 3

ESTIMATION OF PERCENTAGE OF IRON IN THE GIVEN TMT BAR SOLUTION USING STANDARD POTASSIM DICHROMATE SOLUTION

Part A : Preparation of standard solution

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

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

Part B : Estimation of Fe in TMT bar solution

Burette : Standard $K_2Cr_2O_7$ solution

Conical flask : 25 cm^3 TMT bar solution + ¼th test tube conc. HCl, boil + $SnCl_2$ dropwise till colourless. Cool and add ¼th test tube $HgCl_2$

Indicator : $K_3Fe(CN)_6$ (Potassium ferricyanide) - external
End point : Indicator drop fails to develop any colour with the test solution drop

From the volume of $K_2Cr_2O_7$ consumed, calculate the percentage of iron in the given TMT bar solution.