Channabasaveshwara Institute of Technology

(Affiliated to VTU, Belgaum & Approved by AICTE, New Delhi)

(**NAAC Accredited & ISO 9001:2015 Certified Institution**)

NH 206 (B.H. Road), Gubbi, Tumkur – 572 216. Karnataka.

<u>Department of Chemistry</u>

<u>ENGINEERING CHEMISTRY LAB</u>
(BCHEE202/202)

B.E I/II SEMESTER
LAB MANUAL 2023-24

Name: _____

USN: _____   **Branch:** _____

Batch : _____   **Section :** _____

**Prepared and Approved
by Dr. Umesh Laddi
Professor & Head
Department of Chemistry
CIT, Gubbi**

General Chemistry Lab Safety

Conduct:

1. Eating, drinking, and smoking are strictly prohibited in the laboratory.
2. No unauthorized experiments are to be performed.
3. Never taste anything. Never directly smell the source of any vapor or gas; instead by means of your cupped hand, waft a small sample to your nose. Do not inhale these vapors but take in only enough to detect an odor if one exists.
4. Coats, backpacks, etc., should not be left on the lab benches and stools.
5. Always wash your hands before leaving lab.
6. Learn where the safety and first-aid equipment is located. This includes fire extinguishers, fire blankets, and eye-wash stations.
7. Notify the instructor immediately in case of an accident.

Proper Handling of Chemicals and Equipment:

8. Consider **all** chemicals to be hazardous unless you are instructed otherwise. Material Safety Data Sheets (MSDS) are available in lab for all chemicals in use. These will inform you of any hazards and precautions of which you should be aware.
9. Know what chemicals you are using. Carefully read the label *twice* before taking anything from a bottle. Chemicals in the lab are marked with NFPA hazardous materials diamond labels. Learn how to interpret these labels.
10. Excess reagents are **never** to be returned to stock bottles. If you take too much, dispose of the excess.
11. Many common reagents, for example, alcohols and acetone, are highly Flammable. **Do not use them anywhere near open flames.**
12. Always pour acids into water. If you pour water into acid, the heat of reaction will cause the water to explode into steam, sometimes violently, and the acid will splatter.
13. If chemicals come into contact with your skin or eyes, **flush immediately** with copious amounts of water and consult with your instructor.
14. Never point a test tube or any vessel that you are heating at yourself or your neighbour it may erupt like a geyser.
15. Dispose of chemicals properly.
16. Clean up all broken glassware immediately and dispose of the broken glass properly.
17. Never leave burners/hot plates unattended. Turn them off whenever you leave your workstation. Be sure that the gas is shut off at the bench rack when you leave the lab.
18. Beware of hot glass it looks exactly like cold glass.

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Department of Chemistry

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CHEMISTRY DEPARTMENT
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A4.Electroplating of copper on metallic objects

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CIE for the practical component of the Integrated Course :

• On completion of every experiment/program in the laboratory, the students shall be evaluated and marks shall be awarded on the same day. The15 marks are for conducting the experiment and preparation of the laboratory record, the other 05 marks shall be for the test conducted at the end of the semester.

• The CIE marks awarded in the case of the Practical component shall be based on the continuous evaluation of the laboratory report. Each experiment report can be evaluated for 10 marks. Marks of all experiments' write-ups are added and scaled down to 15 marks.

• The laboratory test (duration 02/03 hours) at the end of the 14th /15th week of the semester /after completion of all the experiments (whichever is early) shall be conducted for 50 marks and scaled down to 05 marks.

• Scaled-down marks of write-up evaluations and tests added will be CIE marks for the laboratory component of IPCC for 20 marks.

HOD

A-DEMONSTRATION (ANY TWO) OFFLINE/ VIRTUAL

A1.Synthesis of polyurethane (Online Mode)

Watch the Youtube Video : https://www.youtube.com/watch?v=VIrFRRWG4sA

## A2. Determination of strength of an acid in Pb-acid Battery (Offline Mode):

Determination of Strength of an acid in Pb-Acid battery

Aim: To determine the strength of acid in Lead-Acid battery
Apparatus: Burette, Conical flask, Beaker, Burette stand, Pipette and Wash bottles
Chemicals required: Acid from Lead acid battery, NaOH, Oxalic acid, Phenolphthalein indicator
Principle: Strength of acid in Lead-Acid battery, measure of its ability to neutralize with bases to resist change of pH value of acid due to presence of mineral acids like H_2SO_4.
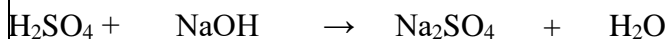
$H_2SO_4 +$ NaOH $\rightarrow$ Na_2SO_4 + H_2O
Procedure:
Standardization of Sodium Hydroxide:
Take 20 ml of oxalic acid solution in to the conical flask and add 2 to 3 drops of Phenolphthalein indicator, resulting the complete solution is colorless. The solution is titrated against with Sodium hydroxide solution. The sample solution changes colorless to pink color. The titration stops and note down the burette value. Then titration should

be repeated until getting concrete readings. Determination of strength of an acid: Take 20 ml of acid solution from lead acid battery in to the conical flask and add 2 to 3 drops of Phenolphthalein indicator, the complete solution is colorless. The solution is titrated against with sodium hydroxide solution. The sample solution changes colorless to pink color. The titration stops and note down the burette value. Then titration should be repeated until getting concrete readings.

Standardization of Sodium hydroxide:

S. No	Volume of sample solution	Burette reading		Volume of oxalic acid
		Initial	Final	

Calculations:

$N_1V_1 = N_2V_2$

Concentration of Sodium Hydroxide (N_1) = ?
Volume of sodium hydroxide   (V_1) =   ml
Concentration of Oxalic acid   (N_2) =   N
Volume of Oxalic Acid   (V_2) =   ml

$$N_1 = \frac{N_2 X V_2}{V_1}$$

Concentration of Sodium hydroxide solution N_1=

1. Strength of acid in Lead-Acid battery

S. No	Volume of sample solution	Burette reading		Volume of Sodium Hydroxide
		Initial	Final	

Calculations:

$N_1V_1 = N_2V_2$

Concentration of Sodium Hydroxide (N_1) =
Volume of sodium hydroxide   (V_1) =   ml
Concentration of Sulphuric acid   (N_2) = ?
Volume of Sulphuric acid   (V_2) =   ml

$$N_2 = \frac{N_1 V_1}{V_2}$$

Concentration of acid in Lead-acid battery N_2=

Result: the strength of acid in Lead –Acid battery is -----------M/lit

B -EXERCISE/EXPERIMENTS

Experiment No. B1   **Date:___/___/ _____**

CONDUCTOMETRIC ESTIMATION OF ACID MIXTRE USING STANDARD NaOH SOLUTION.

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

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

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

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

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

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

The amount of CH_3COOH present in the given solution=   _____g

Calculation:

Normality of NaOH = 1 N

Volume of NaOH required to neutralize HCl, V_1 =---------- cm3

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

$$= \frac{1 \text{ x}}{50}$$

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

The weight of HCl /dm^3 = Normality x Eq.Wt. of HCl

= -----------36.5

= ------------ g

Volume of NaOH required to neutralize CH$_3$COOH, V_2-V_1=

------Cm^3

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

$$= \frac{1 \text{ x}}{50}$$

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

The weight of CH$_3$COOH /dm^3 = Normality x Eq. Wt. of CH$_3$COOH

= -----------60

= ....g.

Volume of NaOH (ml)	Conductance (S/m)
0.0	
0.5	
1.0	
1.5	
2.0	
2.5	
3.0	
3.5	
4.0	
4.5	
5.0	
5.5	
6.0	
6.5	
7.0	
7.5	
8.0	
8.5	
9.0	
9.5	
10.0	
10.5	
11.0	
11.5	
12.0	
12.5	
13.0	
13.5	
14.0	
14.5	
15.0	
15.5	
16.0	
16.5	
17.0	
17.5	
18.0	
18.5	
19.0	
20.0	

Experiment No. B2   **Date: __ /__/ _____**

POTENTIOMETRIC -ESTIMATION OF FAS USING STANDARD $K_2Cr_2O_7$ SOLUTION

Aim: Estimation of potentiometric titration-estimation of FAS using standard $K_2Cr_2O_7$ solution

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

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

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

Where E^o = standard potential of the system.

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

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

Result: Weight of FAS present in 25 cm^3 of the given solution = -------------------- g

Observation and Calculation:

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

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

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

$= \underline{\hspace{4cm}}$

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

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

$= \underline{\hspace{4cm}} g$

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

$= \underline{\hspace{4cm}} g$

Experiment No. B3   **Date: __ /__/ _____**

DETERMINATION OF P^{Ka} OF VINEGAR USING P^H SENSOR

Aim: To determine $_P$ka value of vinegar using P^H sensor.

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

According to Henderson-Hasselbalch equation. $p^H = p^{Ka} + \log$ [salt] / [acid]. Where Ka is dissociation constant of the acid and $p^{Ka} = -\log$ Ka.
      At half equivalence point, [salt] = [acid]. And hence $p^H = p^{Ka}$. Therefore p^H at half equivalence point gives the p^{Ka} of weak acid (Vinegar).

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

Result: The p^{Ka} value of a given weak acid (Vinegar) is ---------------

Observation and Calculation:

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

Equivalence point, X = -----------Cm^3

Half equivalence point, X/2 = ---------Cm^3

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

p^{Ka} of given weak acid (Vinegar) = ---------

Experiment No. B5   Date:___/ ___/ _____

<h3 align="center">Estimation of total hardness of water by EDTA method.</h3>

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

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

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

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

When added to the solution containing metal ions buffered to a pH of about 10, EBT forms a wine red cold complex with the metal ions (see the below structure).
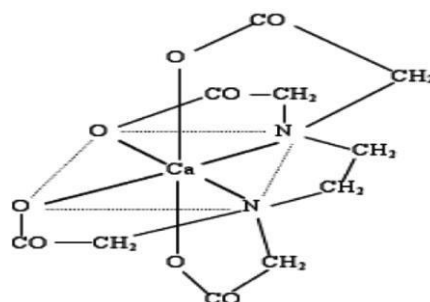
When the solution is titrated with EDTA, EDTA combines with the free metal ions (calcium ions in preference to magnesium ions) present in the solution. At the end point, when all the free metal ions are exhausted, added EDTA dissociates the wine red cold metal-EBT complex, consumes the metal ion and releases the blue cold free indicator. Hence the color changes from red wine to blue.

Procedure:

<u>**Part A: Preparation of standard solution of Disodium salt of EDTA**</u>

Weigh out the given EDTA crystals accurately into a 250ml volumetric flask. Add quarter test tube

of ammonia. Dissolve in distilled water and dilute up to the mark, mix well.

Part B: Determination of total hardness of water sample

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

RESULT: The total hardness of given water sample= ------------- ppm of CaCO$_3$

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

Weight of weighing bottle + EDTA salt (W$_1$) = ------------------g

Weight of empty weighing bottle (W$_2$)   =------------------ g

Weight of EDTA transferred (W$_1$ – W$_2$)   = ----------------- g

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

Part B: Determination of Total hardness of water sample

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

Calculation: Volume of EDTA consumed   =---------------------- ml

1000 cm^3 of 1 M EDTA   = 100 g of CaCO$_3$ (Molecular mass of CaCO$_3$ =100)

$$\text{.........ml of ....M EDTA} = \frac{X \times 100}{1000 \times 1} \text{ g of CaCO}_3 = \text{...}$$

25 cm^3 of the hard water sample contains --------- g of CaCO$_3$

$$10^6 \text{ cm}^3 \text{ of the hard water sample contains} = \frac{X \cdot 10^6}{25} \text{---------------- ppm of CaCO}_3 = \text{----------------------}$$

$$= \text{--------------- ppm of CaCO}_3$$

Thus total hardness of given water sample   = ---------------- ppm of CaCO$_3$

C - STRUCTURED ENQUIRY EXPERIMENTS

Experiment No. C1   **Date: __ /__/ _____**

Estimation of Copper present in electroplating effluent by optical sensor (colorimetry)

Aim: **Estimation of Copper present in electroplating effluent by optical sensor (colorimetry)**

Principle:

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

$$I_o = I_a + I_r + I_t$$

For a glass-air interface I_r is negligible.

$$\text{Therefore, } I_o = I_a + I_t$$

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

$$A = \log I_o / I_t = \text{€}Ct$$

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

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

A series of std. solutions of copper 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 solution is measured at 620 nm, since the complex shows maximum absorbance at this wavelength. The absorbance values are plotted against volume of copper sulphate to get a calibration curve.

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

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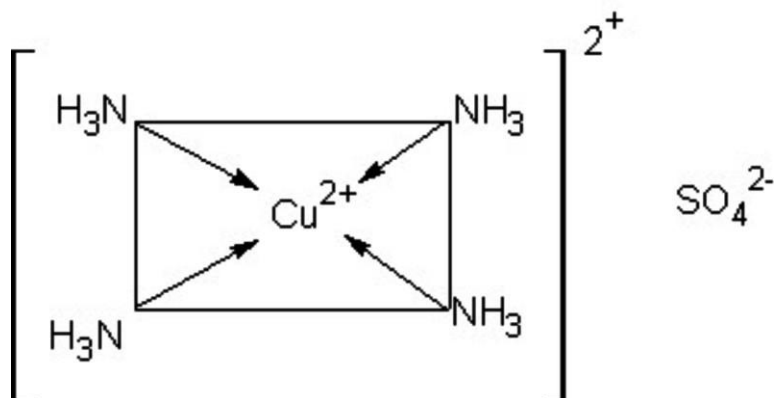
Procedure:

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

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

Observation and calculation:

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

From graph, volume of the given **Electroplating Effluent** , a =_____ml
Copper present in the given **Electroplating Effluent** solution = 2.037mg x volume of test solution ('a' ml)

$$= 2.037 \text{ mg x } \underline{\hspace{3cm}}$$

$$= \underline{\hspace{4cm}} \text{mg}$$

Experiment No. C2   **Date:___/___/ _____**

Determination of Viscosity coefficient of lubricant (Ostwald's viscometer)

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

Principle:

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

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

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

$$\eta_L = \frac{\eta_w \times t_L \times d_L}{t_w \times d_w}$$

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

Procedure:

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

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

Observation and calculation:
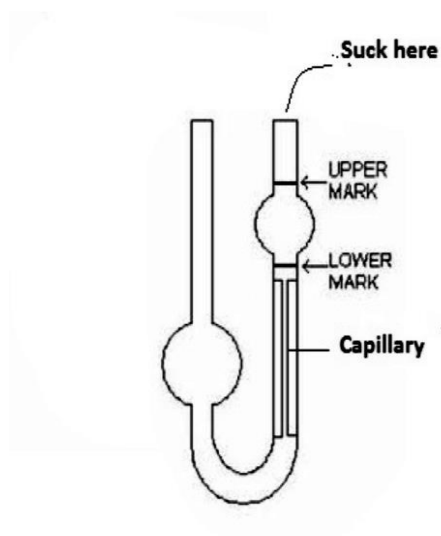
	Trail-1 (sec)	Trail-2 (sec)	Trail-3 (sec)	Average (sec)
Lubricant (t_L)				
Water (t_w)				

Laboratory Temperature = ……………..

η_w = Viscosity coefficient of water   = …… millipoise

η_L = Viscosity coefficient of lubricant   = ?

d_w = density of water   = ...g/cc

d_L = density of lubricant   = ...g/cc

$$\eta_L = \frac{\eta_w \times t_L \times d_L}{t_w \times d_w}$$

$$= \frac{\text{X} \qquad \text{X}}{\text{X}}$$

$$= \overline{\qquad\qquad\qquad}$$

=.....................millipoise.

The coefficient of viscosity of the given lubricant = ...millipoise.
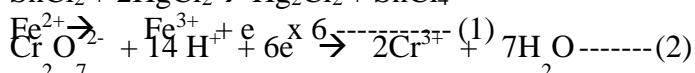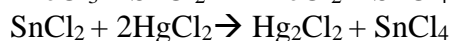
Experiment No. C3   Date: __ /__/ _____
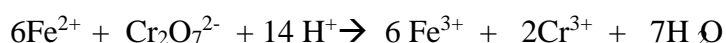
Estimation of iron in TMT bar by diphenyl amine/external indicator method.

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

Principle: Corrosion is a natural process that converts a refined metal into a more chemically stable form such as oxide, hydroxide, carbonate or sulfide (entropy zero). Interestingly, the term 'rusting' is generally used to refer to the corrosion of objects made of iron or iron-alloys. It is the gradual destruction of materials (usually a metal) by chemical and/or electrochemical reaction with their environment. Rusting causes iron to become flaky and weak, degrading its strength, appearance and permeability. Rusted iron does not hold the desirable properties of iron. The rusting of iron canlead to damage to automobiles, railings, grills, and many other iron structures. It's important to know how much iron is lost due to rusting i.e., the formation of the rust ($Fe_2O_3.3H_2O$). To estimatethe rate of corrosion, one of the common method is the estimation of amount of iron present in therust. To estimate the amount of iron in rust, the rust is dissolved in concentrated HCl. Iron is presentin the rust solution as Fe^{3+} ions and is reduced to Fe^{2+} ions using stannous chloride in hot conditionin presence of concentrated HCl, because Fe^{+3} can't react with $K_2Cr_2O_7$. The excess of stannous chloride added is oxidized by treating the solution with mercuric chloride. The ferrous ions in the resulting solution are titrated against the standard potassium dichromate using potassium ferricyanide as external indicator. Potassium ferricyanide produces an intense blue color with ferrous ions due to the formation of ferric ferricyanide complex[$4FeCl_3$ + $3K_4Fe(CN)_6 \rightarrow$ $Fe_4[Fe(CN)_6]_3 + 12KCl)$] .

$Fe_2O_3.3H_2O + 6HCl \rightarrow 2FeCl_3 + 6H_2O$
$2FeCl_3 + SnCl_2 \rightarrow 2 FeCl_2 + SnCl_4$
$SnCl_2 + 2HgCl_2 \rightarrow Hg_2Cl_2 + SnCl_4$
$Fe^{2+} \rightarrow Fe^{3+} + e$ x 6 ------- (1)
$Cr_2O_7^{2-} + 14 H^+ + 6e \rightarrow 2Cr^{3+} + 7H_2O$ -------(2)
Adding (1) & (2)

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

Procedure:

Part A:Preparation of standard $K_2Cr_2O_7$ solution
Weigh out 0.5g of $K_2Cr_2O_7$ crystals accurately and transfer into a 250ml volumetric flask. Dissolve in distilled water and dilute up to the mark, mix well.

Part B:Estimation of Iron
Pipette out 25 cm^3 of the rust solution into a clean conical flask. Add a quarter test tube of concentrated HCl and heat the solution to boiling. Add stannous chloride to the hot solution drop wise till the yellow solution turns colorless. Add 2 more ml drops to ensure complete reduction. Cool and add a quarter test tube of mercuric chloride (silky white ppt, mercurous chloride, is formed). Add one test tube of water. Place number of drops of freshly prepared potassium ferricyanide indicator on the wax paper. Add a small quantity of $K_2Cr_2O_7$ from burette to the conical flask containing rust solution and mix well. Take a drop of the solution from the conical flask and bring it in contact with a drop of the indicator on wax paper. The color of the indicator turns blue. Continue the titration by adding increment of 1cm^3 of $K_2Cr_2O_7$ at a time and test till a drop of reaction mixture fails to produce any color with the indicator (Note: Clean the glass rod after every

test).Repeat the titration by taking another 25 cm^3 of the rust solution. This time add most of the K$_2$Cr$_2$O$_7$ solution required at a stretch and then drop wise. Mix the contents of the flask after every addition and test a drop of the titrated mixture with a drop of the indicator as described above till the color of the indicator drop does not change. Repeat for concordant values.

RESULT: Percentage of iron in the given rust solution = ----------------------

Observation: **Part A:Preparation of standard K$_2$Cr$_2$O$_7$ solution**

Weight of weighing bottle + K$_2$Cr$_2$O$_7$ crystals (W$_1$)   = --------- ------- g
Weight of empty weighing bottle (W$_2$)   =------------------ g
Weight of K$_2$Cr$_2$O$_7$ crystals transferred (W$_1$ – W$_2$)   =------------------ g

$$\text{Normality of K}_2\text{Cr}_2\text{O}_7 = \frac{\text{Weight of K}_2\text{Cr}_2\text{O}_7(W_1 - W_2) \times 4}{\text{Equivalent weight of K}_2\text{Cr}_2\text{O}_7 (49)} = \text{....................} N$$

Part B:Estimation of Iron :

Burette   : Std. K$_2$Cr$_2$O$_7$ Solution
Conical Flask : 25 ml rust solution + 1/4th test tube of con. HCl, boil +
                      SnCl$_2$ drop wise till colorless. Cool & add 1/4th test tube of HgCl$_2$
Indicator   : Potassium ferricyanide as external indicator.
Color Change : No change in the color of indicator (due to the absence of ferric ions)

Burette reading	I	II	III
Final burette reading			
Initial burette reading			
Volume of K$_2$Cr$_2$O$_7$ run down in cm^3			

Calculation:

Volume of K$_2$Cr$_2$O$_7$ consumed   = -------------------

Weight of Rust in 25 cm^3 = 0.1g
1000 cm^3 of 1N K$_2$Cr$_2$O$_7$ ≡ 55.85 g of Fe(1 equivalent of Fe)

--------cm^3 ------*N* K$_2$Cr$_2$O$_7$ ≡ 55.85 x   x

$$= \frac{}{1000 \text{ x1}}$$

≡---------- g of Fe / 25 Cm^3 of rust solution

Percentage of iron in the given rust solution =   g x 100

$$\frac{}{\text{Weight of rust (0.1)}} = \text{- - - - - - - - - -}$$

$$= \underline{\qquad}$$

Experiment No. C5   Date:____/____/ _____

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

Aim: To determine the COD of waste water.

Principle:

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

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

Procedure:

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

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

Back titration:

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

Blank Titration:

Pipette out 10ml of standard potassium dichromate solution. Add 30ml 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 color turns from bluish green to reddish brown.

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

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

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

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

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

Calculation:

Volume of FAS consumed in Back titration = --------------------$c x m^3$ ($Y \, cm^3$)

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

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

$1000 \, Cm^3$ 0f 1 N FAS solution = 1 equivalent of oxygen = 8×10^3 mg of oxygen

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

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

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

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

$1000 \, cm^3$ of waste water sample   = $\dfrac{X \quad 1000}{25}$ mg of oxygen = ---------------

$$= \text{----------------------} \text{ mg of oxygen/} dm^3$$

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

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

D– OPEN ENDED EXPERIMENTS (ANY TWO)

D1: Evaluation of acid content in beverages by using pH sensors and simulation.
D2. Construction of photovoltaic cell.
D3. Design an experiment to Identify the presence of proteins in given sample.
D4. Searching suitable PDB file and target for molecular docking

LIST OF EQUIPMENTS

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

POTENTIOMETER with built-in magnetic stirrer & standard cell

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

Experiment: Estimation of Copper colorimetrically.

The advantages of the Digital Photoelectric Colorimeter 112 at a glance:

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

Experiment: Estimation of Acids in acid mixture conductometrically.

CONDUCTIVITY METER with inbuilt magnetic stirrer & standard conductance

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

Experiment: Determination of pKa of weak acid using pH meter

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

VIVA QUESTIONS WITH ANSWERS

B1.CONDUCTOMETRIC ESTIMATION OF ACID MIXTRE USING STD. NaOH SOLUTION.

1. What is conductivity of an electrolyte solution?
Is a measure of its ability to conduct electricity?

2. What is conductance?
    Reciprocal of resistance is called conductance.

3. What is a cell?
    A device, which produces an EMF and delivers electric current as the results of a chemical reaction.

4. What factors determine the conductance of a solution ?
    Two factors determining the conductance of a solution are;
    1. Mobility of ions (Higher the mobility, higher is the conductance) and
    2. Number of ions (M the number of ions, m is the conductance).

5. What is cell constant?
Is a function of the electrode areas, the distance between the electrodes and the electrical field pattern between the electrodes. Cell constant is defined as the ratio of the distance between the electrodes, d, to the electrode area, A.

6. What is the unit of cell constant?
     Commonly, expressed in cm^{-1},

7. State Ohm's law.
    Current, I (ampere), flowing in a conductor is directly proportional to the applied electromotive force, E (volt) and inversely proportional to the resistance R (ohm) of the conductor. $I = E/R$.

8. What is the unit of conductance?
    $Ohm^{-1}m^{-1}$ or Sm^{-1} (S- siemens)**.**

9. Mentions the different types of conductance.
     i) Specific conductance.
     ii) Equivalent conductance
    iii) Molar conductivity.

10. Which of the above conductivity is measured during the conductometric titration ?
    Specific conductance.

11. What is specific conductivity?
    Conductivity of a solution between $1cm^2$ area and kept 1 cm apart.

12. What is equivalent conductance?
    Conductivity of a solution, which contains 1g equivalent of solute, when placed between 2 electrodes of 1cm apart. $\lambda = kV$.

13. What is molar conductivity?

Conductivity of the solution, which contains 1g molecular weight of a substance, when placed between two electrodes of 1 cm apart, $\mu = kV$.

14. What is the principle involved in conductometric titration?
There is a sudden change in conductance of a solution near the end point. Hence the end point is determined graphically by plotting conductance against the titer values.
Measuring resistance and the dimension of conductivity cell (Ohm law) and plotting the titration curve (conductance-as reciprocal of resistance against the volume of titrant) is the principle.

15. Explain the variation in conductivity at the end point.
In the case of NaOH and HCl experiment, addition of NaOH to HCl decreases the conductance because of the replacement of high conducting mobile H^+ ions (Conductivity : 350 $Ohm^{-1}m^{-1}$) by the less mobile Na^+ ions (Conductivity : 50 $Ohm^{-1}m^{-1}$). Trend continues till all the hydrogen ions are replaced and end point is reached. Further addition of NaOH, increases the conductance because of the presence of free OH^-ions (Conductivity : 198 $Ohm^{-1}m^{-1}$).

16. Why are the slopes of curves before and after neutralization point not exactly the same ?
Before the neutralization point, the conductivity is mainly due to the highly mobile H^+ ions and after the end point, it is mainly due to OH^-ions, which are less mobile compared to H^+ ions. As a result, magnitude of decrease in conductivity is m before the neutralization point compared to that after the neutralization point.

17. How is the accuracy of the method determined?
Accuracy of the method is greater when the angle of intersection is m acute and m nearly the end points of the graph on a straight line.

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

B2. Potentiometric estimation of FAS using $K_2Cr_2O_7$

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

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

3. What is meant by emf?
EMF is potential difference required to drive current across the electrodes.
$EMF = E_{cathode} - E_{anode}$

4. What is potentiometer?
It is a device or circuit used for measuring the emf between the two electrodes

5. What is potetiometric titration?
   It is the measurement of emf to determine the concentration of ionic species in solution is called potentiometry

6. What is the principle of potentiometric titration?
   The measurement of the emf between two electrodes (indicator and reference) and to determine the equivalence point of redox reaction

7. Which are the electrodes used in potentiometric electrode?
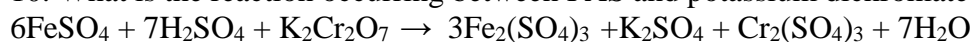Platinum electrode as indicator electrode (anode) and calomel electrode as reference electrode (cathode)

8. What is determining factor in the oxidation-reduction reaction?
   The determining factor is the ratio of the concentrations of the oxidized and reduced forms.

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

10. What is the reaction occurring between FAS and potassium dichromate?
$6FeSO_4 + 7H_2SO_4 + K_2Cr_2O_7 \rightarrow 3Fe_2(SO_4)_3 + K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O$
   Acidified potassium dichromate oxidizes ferrous sulphate to ferric sulphate and itself gets reduced to chromic sulphate

11. Why sulphuric acid is added to FAS solution during emf measurement?
   In presence of sulphuric acid , potassium dichromate oxidizes ferrous ion(Fe^{+2}) to ferric ion (Fe^{+3})

12. Why the color of the solution changes to green on adding potassium dichromate?
Due to formation of green coloured chromic sulphate.

13. Why emf rapidly change at the equivalence point?
   At the equivalence point, emf depends on concentration of both ferric ion and dichromate ion .There of emf rapidly change at the equivalence point.

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

B3. Determination of pKa of vinegar using pH sensor (Glass electrode)

1. How is p^{Ka} of a weak acid evaluated from the graph?
   pH at half equivalence point gives the pKa of a weak acid

2. What is a weak acid?
Weak acids are weak electrolytes, which ionize incompletely in aqueous solution.

3. Give an example for weak acid?
   Acetic acid, Oxalic acid

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

pKa is defined as, negative logarithm to the base 10 of dissociation constant of weak acid. i.e.,
pKa = - $\log_{10}$Ka, Where Ka is the dissociation constant of the weak acid.

5. What is meant by p^{H} ?

pH is defined as negative logarithm to base 10 of hydrogen ion concentration. p^{H} = - $\log_{10}[H^{+}]$

6. What is the modern definition (IUPAC) of p^{H}?

The difference in pH between two solutions S (std) and X (unknown) at the same temperature
with the same reference electrode and with hydrogen electrodes at the same hydrogen pressure
is given by

$$pH\,(X) - pH\,(S) = \frac{E_X - E_S}{2.3026\ RT / F}$$

7. Why glass electrode is called an ion selective electrode?

Because it is able to respond to certain specific ions (H^{+}ions) only and develop a potential
While ignoring the other ions in a solution.

8. How is the measurement of p^{H} made?

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

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

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

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

At half equivalence point concentration of salt = concentration of acid, Theref p^{H} = p^{Ka}.

11. How are p^{Ka} and strength of a weak acid related?

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

12. What are the electrodes used in the measurement of pH and determination of p^{Ka}?

Glass electrode (indicator electrode) and calomel electrode (reference electrode).
In this case, glass electrode acts as anode & calomel acts as cathode.

13. Why is pH increases suddenly after the equivalence point?

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

B5. Estimation of total hardness of water by EDTA method.

1. **Why is ammonia ammonium chloride buffer added?**
   Ammonia ammonium chloride is added to maintain a pH of 10. Otherwise pH decreases as
   H^{+} ions are released due to the substitution of metal ions for H^{+} ions in EDTA.

2. **What is hard water?**
   Hard waters are a kind of water that requires considerable amounts of soap to produce foam
   and lather.

---

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

4. How are the waters classified based on the degree of hardness?
   Soft water   0 to 75 mg / litre
   Moderately hard water   75 to 150 mg / litre
   Hard water   150 to 300 mg / litre
   Very hard water   > 300 mg / litre

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

6. What is the difference between temporary and permanent hardness?
   Temporary hardness is due to unstable bicarbonates of Ca and Mg, while permanent hardness
   is due to m stable Cl^- and SO_4^{2-} of Ca and Mg

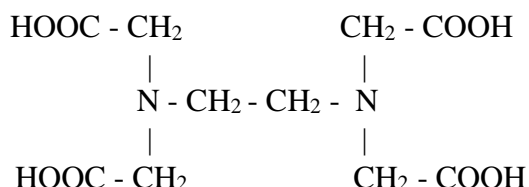**7. How temporary hardness is be removed?**
   The temporary hardness of water can be removed by boiling water during which bicarbonates
   decomposes to give carbonates.

8. How do you express the total hardness of water?
   It is expressed as parts per million of $CaCO_3$.

9.What is EDTA?
   Ethylene diamine tetra acetic acid.

10. Write a structural formula of EDTA.

$$HOOC - CH_2 \qquad\qquad CH_2 - COOH$$
$$| \qquad\qquad\qquad\quad |$$
$$N - CH_2 - CH_2 - N$$
$$| \qquad\qquad\qquad\quad |$$
$$HOOC - CH_2 \qquad\qquad CH_2 - COOH$$

11. How many replaceable hydrogen atoms are present in disodium salt of EDTA?
Two.

12. Why is disodium salt of EDTA preferred to EDTA?
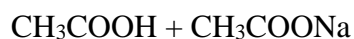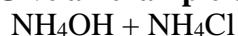   Because EDTA is sparingly soluble in water.

13. Why is ammonia solution added while preparing EDTA solution?
   Because to increase the rate of dissolution of EDTA salt.

14. What is buffer capacity?
  The capacity of a solution to resist the change in pH

15. Give an example of acidic buffer?

CH$_3$COOH + CH$_3$COONa

16. Give an example of basic buffer?
NH$_4$OH + NH$_4$Cl

17. What is reserve alkalinity?
Buffer solutions are considered to possess reserve acidity as well as alkalinity.
According to Henderson-Hasselbalch equation, pH = pKb + log$_{10}$ [salt] / [base],
At half equivalence point [Concentration of salt = Concentration of base],
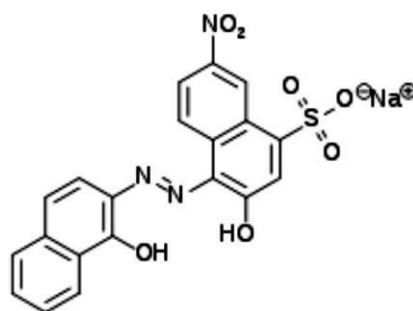Theref pH = pKb. Similarly, for reserveacidity for acidic buffer.
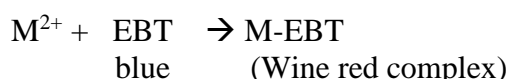
18. What is buffer solution?
The solution which resists change in its pH value even after adding small amounts of an acid or base to it.

19. What is the chemical name of EBT ?
Sodium 4-[(1-hydroxynaphthalen-2-yl-hydrazinylidene]-7-nitro-3-oxo-Y-naphthalene-1-sulfonate.

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

M^{2+} + EBT   → M-EBT
        blue   (Wine red complex)

M^{2+} + EDTA   → M-EDTA   +   EBT (Blue)
                    (Stable complex)   (protonated)

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

21. Titrations involving EDTA carried out slowly towards the end point. Justify?
Because the rate of formation of the metal complex of EDTA is very slow.

22. What is the application of hardness data in environmental engineering practice?
i) Hardness of water is an important consideration in determining the suitability of a water for domestic and industrial uses.
ii) Determination of hardness serves as a basis for routine control of softening processes.

C1. Estimation of Copper present in electroplating effluent by optical sensor (colorimetry)

1. What is the basic principle of colorimetric measurements?
   It consists of comparing under well defined conditions, the color produced by the substances in amount with the same colour produced by a known amount of material being determined.

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

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

4. What is photoelectric colorimeter?
    It is an electrical instrument, which measures the amount of light absorbed using a photocell.

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

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

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

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

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

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

11. State Beer-Lambert's law.
     The amount of light absorbed is directly proportional to the concentration of the solution.

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

12. What is calibration curve?

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

13. What is meant by transmittance?

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

$T = I_t / I_0$

Absorbance $= 1 / T = I_0 / I_t$

14. What is absorbance or optical density?

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

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

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

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

16. What is a stock solution?

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

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

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

18. Mention a few advantages of photoelectric colourimetric determinations.

   i) A colourimetric method will often give m accurate results at low concentrations than the corresponding titrimetric or gravimetric procedure.
   ii) A colorimetric method may frequently be applied where no satisfactory gravimetric or titrimetric procedure exits i.e. for certain biological substance.

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

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

20. What is blank solution?

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

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

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

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

Cuprammonium sulphate, [Cu $(NH_3)_4$]SO_4 a dark blue color complex
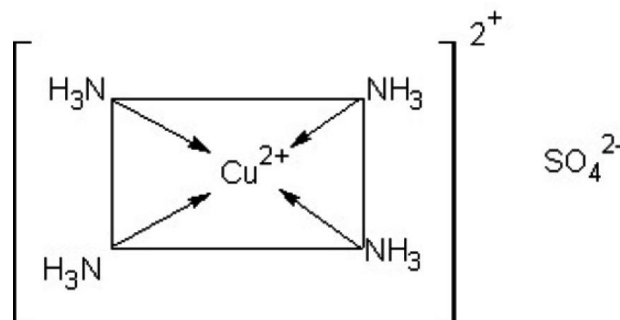
23. Why is ammonia added? Why is that same amount of ammonia added?
Ammonia is added to get cuprammonium sulphate $[Cu(NH_3)_4]SO_4$ a dark blue complex. Same amount of ammonia is added to nullify the absorbance due to any colouring impurities present in ammonia.

24. Why is estimation of copper done at 620 nm wave length?
The estimation of copper is carried at 620 nm wave length because the complex shows maximum absorbance at 620 nm.

25. Mention a few important criteria for a satisfactory colorimetric analysis.
i) In order to obey Beer-Lambert's law, the solute must not undergo salvation, association, dissociation, hydrolysis or polymerization in the solvent used.
ii) The color produced should be sufficiently stable to permit an accurate reading to be taken.
  iii) Clear solutions free from traces of precipitate or fign substances in either blank or standard test solution should be used.

C2. Determination of Viscosity coefficient of lubricant (Ostwald's viscometer)

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

2. What is viscosity co-efficient of a liquid?
The viscosity coefficient of a liquid is defined as the tangential force per unit urea required to maintain a unit velocity gradient between any two successive layers of a liquid situated unit distance apart.

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

4. The density of a substance is expressed relative to what?
The density of a substance is expressed relative to that of water at 4^0C

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

6. How are specific gravity and density of liquid related?
Density of liquid = specific gravity of the liquid x Density of water at the same temperature

7. What is the SI unit of viscosity coefficient?
Pascal second (1 ps = 1 kg m^{-1} s$^-$)

8. What is the unit of viscosity coefficient in cgs system?
Millipoise.
9. What are the factors that affect the viscosity of a liquid?
  i) Increase in molecular weight results in an increase in viscosity.
  ii) Branched chain compounds have higher viscosity than those involving straight chain compounds.

iii) The polar compounds are m viscous than the non polar ones. The presence of hydrogen bonds cause the viscosity to increase.

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

v) Temperature has marked influence on the viscosity of a liquid (about 2% per degree).

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

Poiseuille law,   Where P = hgd

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

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

ρ = pressure difference between two ends of the tube,

η = the coefficient of viscosity of the liquid,

t = time required for the volume.

11. How does viscosity vary with temperature?

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

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

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

13. Why is acetone used for cleaning viscometer?

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

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

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

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

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

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

Viscosity of a liquid is inversely proportional to its mobility.

17. What is fluidity of a liquid?

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

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

C3. Estimation of iron in TMT bar by diphenyl amine method

1.What is meant by TMT bar?

TMT stands for 'Thermo Mechanical Treatment' that is also known as metallurgical process. These TMT Bars are manufactured under the IS (Indian Standards) Standards.

2. Is TMT bar steel or iron?

Known for its high-strength, sturdy built and robust appearance with the capability to provide better reinforcement to any concrete structure, TMT (Thermo Mechanically Treated) Steel Bars are ideal for building earthquake-resistant structures.

**3. Why nitric acid (50-70%) is added to dissolve TMT bar crushed, along with water (30-50%)?**

The solution should be made by slowly adding the nitric acid to the water in an acid-resistant container. Create enough of this solution to fully submerge the steel you are trying to dissolve. The iron in steel, producing iron nitrate and hydrogen gas.

4. What is the role of stannous chloride?

Stannous chloride reduces Ferric ion to ferrous ion and a slight excess is added to ensure complete reduction. $2FeCl_3 + SnCl_2 \rightarrow 2\ FeCl_2 + SnCl_4$

5. Why is mercuric chloride added?

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

$$SnCl_2 + 2HgCl_2 \rightarrow Hg_2Cl_2 + SnCl_4$$
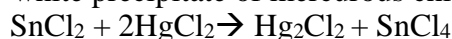
6. What happen when excess of stannous chloride is not removed?

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

**7.Name the external indicator used in iron estimation.**

Potassium ferri cyanide.

8. What is the end point color change during the external indicator, Potassium Indicator?

The test solution fails to induce any color, due to the absence of Ferrous ion and hence the colour is either that of light colour of $K_2Cr_2O_7$ (Dry grass).

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

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

$$3\ Fe^{2+} + 2\ K_3\ [Fe(CN)_6] \rightarrow Fe_3\ [Fe(CN)_6]_2 + 6K^+$$
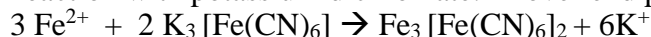
10. Why potassium Ferro cyanide cannot be used as an indicator in the estimation of Fe

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

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

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

$$6Fe^{2+} + Cr_2O_7^{2-} + 14\ H^+ \rightarrow 6\ Fe^{3+} + 2Cr^{3+} + 7H_2O$$
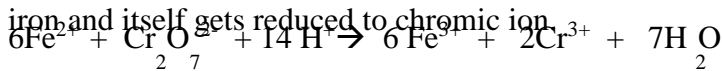
12. Name the internal indicators used for iron determination?

Ex: a) Diphenyl amine (color change will be from colourless to blue-violet colour The diphenylamine (I) undergoes oxidation first into a colourless diphenylbenzidine (II) which is the real indicator and is reversibly further oxidized to diphenylbenzidine violet (III).
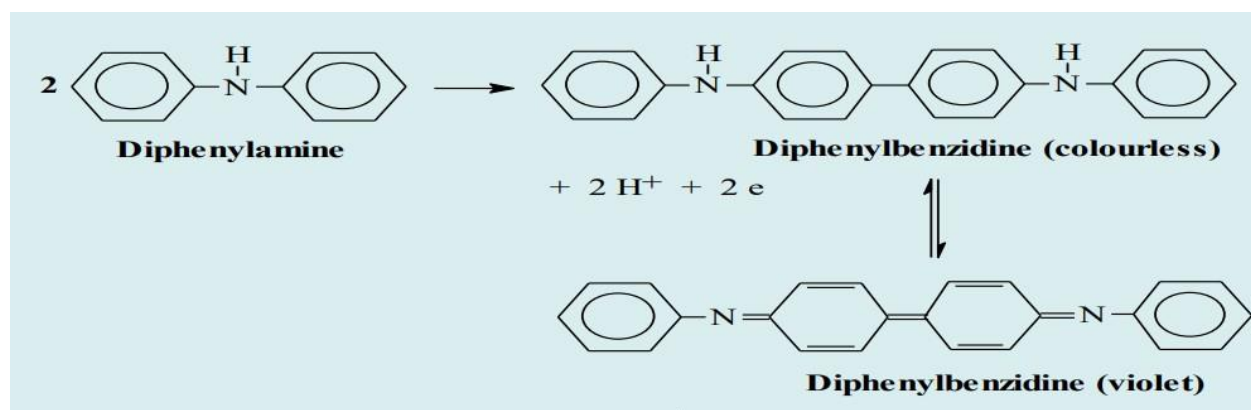
b) N-phenylanthranilic acid (from greenish to purple- Indicator is not oxidized as long as Fe2+ ions are there in the solution. The slight excess amount of dichromate will oxidize the indicator when all of the Fe2+ ions have been converted to Fe3+ ions resulting in colour change of the solution from greenish (due to Cr3+) to purple.

C5. Determination of Chemical Oxygen Demand (COD) of industrial waste water sample

1. What is sewage?

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

2. What is meant by industrial sewage?

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

3. What is COD?

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

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

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

5. **What is BOD**?

It is the amount of oxygen required for the complete oxidation of organic matter present inliter of waste water over period of 5 days at 20^0 C.

6. Why COD greater than BOD value?

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

7. What are end products of oxidation?

CO_2 & H_2O.

8. Why H_2SO_4 is added to conical flask?

Because Potassium dichromate acts as an oxidizing agent in presence of H_2SO_4.

9. What is the unit of COD?

mg/litr of O_2 or mg/dm^3 of O_2 or ppm of O_2.

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

Potassium di chromate.

11. Explain the colour change during the titration.

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

Ferroin (red)   Ferrin (blue)
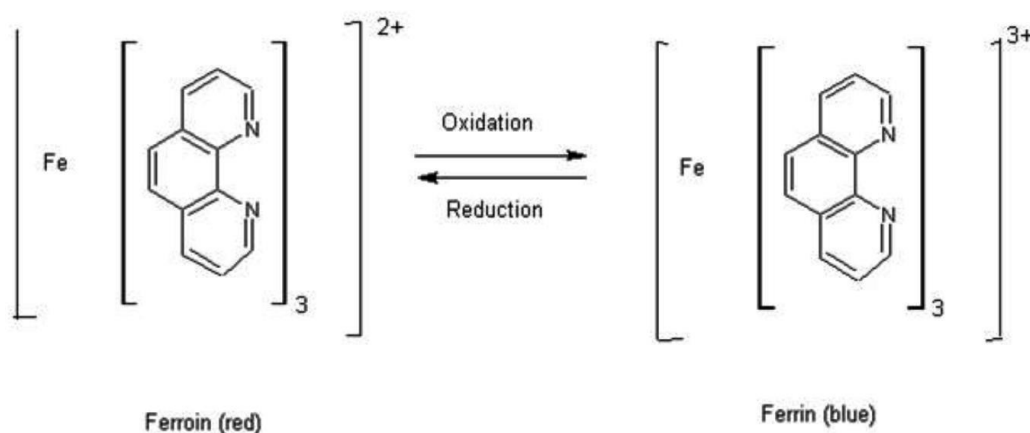
Active ingrediant ions

12. What is back titration?

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

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

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

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

Aromatic hydrocarbons and pyridine are not oxidized in COD test.

15. What is the role of silver sulphate?

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

16. What is the role of mercuric sulphate?

Chloride ions normally present in high concentration in waste water undergo oxidation in
COD test and cause erroneously high results. Mercuric ions of mercuric sulphate bind the

halide ions present in waste water to form poorly ionized mercuric chloride and prevent the precipitation of silver halide by making halide ions unavailable.

17. If nitrites are present as impurities, what is the reagent used?
Sulphamic acid (amido sulphonic acid, H_3NSO_3)

18. What are the products formed after COD analysis?
During COD analysis organic matter is completely oxidized to carbon dioxide and water by acidified potassium dichromate solution.

19. **Why is sulphuric acid added during the preparation of std. FAS solution?**
Sulphuric acid is added to prevent the hydrolysis of ferrous sulphate into ferrous hydroxide.

20. What is the composition of ferroin?
Ferroin is ferrous 1, 10-Phenanthroline sulphate.

Ferroin indicator
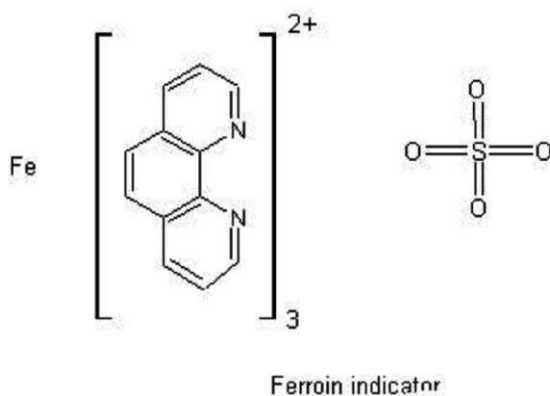
21. Why blank titration is necessary?
Because the COD measures the oxygen demand in the sample of water, it is important that no outside organic material be accidental added to the sample to be measured. To control for this, so called blank sample is required.

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

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

MODEL PROCEDURES

B1: CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE USING STD. NaOH SOLUTION.

Pipette out 50ml of acid mixture in to a beaker. Immerse the conductivity cell in to it & connect it to conductivity meter and measure the conductance. Add NaOH from the burette in the increments of 0.5 ml and measure the conductance after each addition.Plot a graph of conductance against volume of NaOH & determine the equivalence point. Calculate the normality & weight of HCl & CH_3COOH in the given solution.
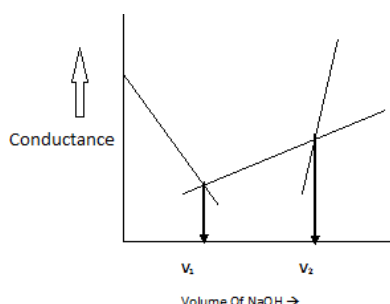

Where, V_1= Volume of NaOH required to neutralize HCl.
        (V_2-V_1) = Volume of NaOH required to neutralize acetic acid.

B2. Potentiometric estimation of FAS using $K_2Cr_2O_7$.

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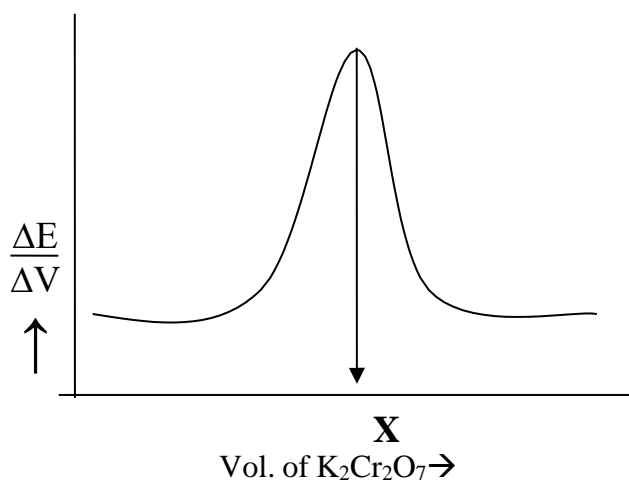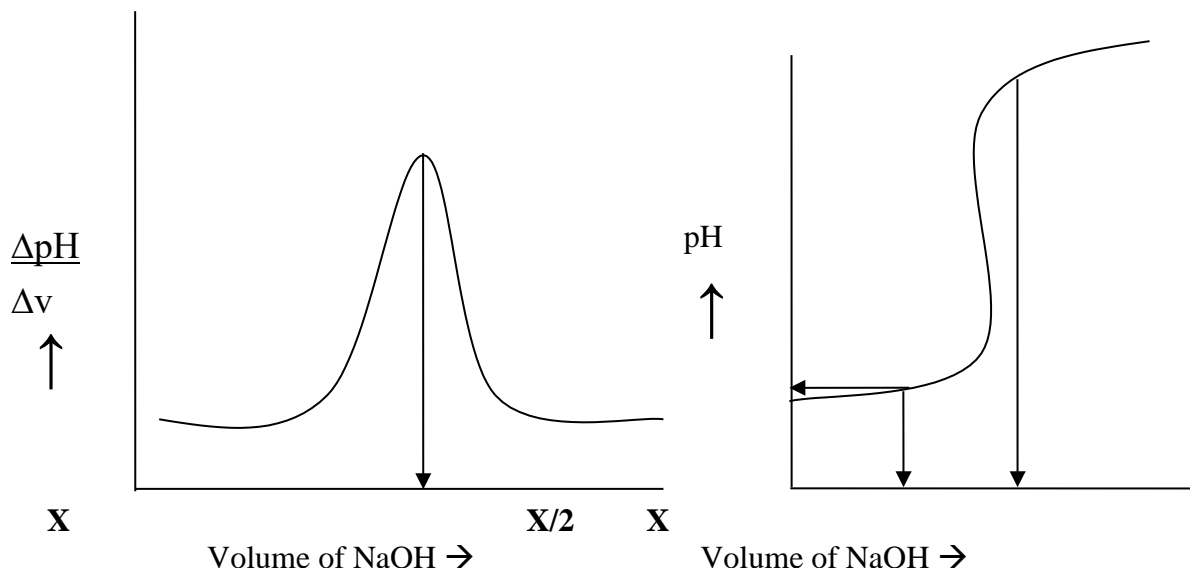

Where, X = Equivalence point

B3.Determination of pKa of vinegar using pH sensor (Glass electrode).

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

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

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

B5. Estimation of total hardness of water by EDTA method.

Part A: Preparation of standard solution

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

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

#### Part B: Determination of total hardness

Burette   : Standard EDTA Solution.

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

Indicator   : Eriochrome black-T.

Colour Change   : Wine red to blue.

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

C1. Estimation of Copper present in electroplating effluent by optical sensor (colorimetry)

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

O
D

↑

X
Vol. of CuSO$_4$→
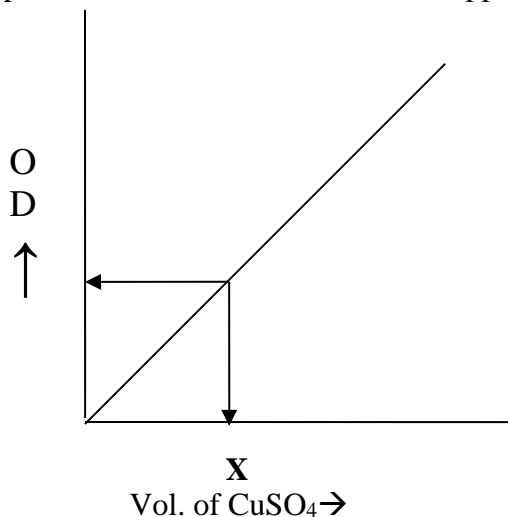
X – volume of test solution

C2. Determination of Viscosity coefficient of lubricant (Ostwald's viscometer)

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

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

η_w = Viscosity coefficient of water
η_L = Viscosity coefficient of liquid
d_w = density of water
d_L = density of liquid
t_l= time flow of liquid and t_w = time flow of water.

C3. Estimation of iron in TMT bar by diphenyl amine method

Part A:Preparation of standard K$_2$Cr$_2$O$_7$ solution
Weigh out the given K$_2$Cr$_2$O$_7$ crystals accurately into a 250ml volumetric flask. Dissolve in distilled water and dilute up to the mark, mix well.

$$\text{Normality of K}_2\text{Cr}_2\text{O}_7 = \frac{\text{Weight of K}_2\text{Cr}_2\text{O}_7 \text{ taken X 4}}{\text{Equivalent weight of K}_2\text{Cr}_2\text{O}_7 \text{ (49)}}$$

Part B:Estimation of Iron in rust

Burette   : Standard $K_2Cr_2O_7$ Solution

Conical Flask   : 25 ml Rust solution + $1/4^{th}$ test tube of con. HCl, boil +

$SnCl_2$ drop wise till colorless. Cool & add $1/4^{th}$ test tube $HgCl_2$

Indicator   : Potassium ferricyanide

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

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

C5. Determination of Chemical Oxygen Demand (COD) of industrial waste water sample.

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

Weigh out the given FAS crystals accurately into a 250 ml volumetric flask. Add 2 test tube of

H_2SO_4. Dissolve in distilled water and dilute up to the mark, mix well.

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

Part B: Determination of COD of waste water sample.

Burette   : Standard FAS Solution.

Conical Flask   : 25 ml waste water sample +10 ml $K_2Cr_2O_7$+ 1 test tube of 1:1 H_2SO_4

Indicator   : Ferroin

Colour Change   : Blush green to reddish brown.

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

Reference Books:

1.G.H.Jeffery,J.Bassett,J.MendhamandR.C.Denney,**"Vogel'sTextBookofQuanti tativeChemicalAnalysis"**

2.O.P.Vermani&Narula,**"TheoryandPracticeinAppliedChemistry"**,New Age International Publisers.

3.GaryD. Christian, **"Analyticalchemistry**", 6[th]Edition, WileyIndia.

General Instructions for Volumetric Analysis