



# ***ENGINEERING CHEMISTRY LABORATORY MANUAL***

***I/II Semester (18CH1ILCHY/18CH2ILCHY)  
2018-19***



Name of the Student :

Semester /Section :

USN :

Batch :

**DAYANANDA SAGAR COLLEGE OF ENGINEERING**

**Accredited by National Assessment & Accreditation Council (NAAC) with 'A' Grade**  
(An Autonomous Institution affiliated to Visvesvaraya Technological University, Belagavi & ISO 9001:2015 Certified)

**DEPARTMENT OF CHEMISTRY  
SHAVIGE MALLESWARA HILLS, KUMARASWAMY LAYOUT  
BENGALURU-560078**

## **Vision of the Institute**

To impart quality technical education with a focus on Research and Innovation emphasizing on Development of Sustainable and Inclusive Technology for the benefit of society.

## **Mission of the Institute**

- ❖ To provide an environment that enhances creativity and Innovation in pursuit of Excellence.
- ❖ To nurture teamwork in order to transform individuals as responsible leaders and entrepreneurs.
- ❖ To train the students to the changing technical scenario and make them to understand the importance of Sustainable and Inclusive technologies.



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**DEPARTMENT OF CHEMISTRY, BENGALURU-560078**

**VISION OF THE DEPARTMENT**

To be a role model to serve the Nation's needs by encouraging the study of Chemistry as a means for improving the condition of mankind by ameliorating his environment

**MISSION OF THE DEPARTMENT**

- To adopt the best pedagogical methods in order to maximize knowledge transfer.
- To have adequate mechanisms to enhance understanding of Implementation of theoretical concepts in practical scenarios.
- To carry out high quality research leading to the creation of knowledge and of intellectual property.
- To provide the best facility, infrastructure, and environment to its students, researchers and faculty members, creating an ambience conducive for excellence in technical education and research.
- To impart professional ethics and an approach towards inter- disciplinary thinking by providing them with educational and research opportunities between Chemistry and other fields of study

**COURSE OBJECTIVES**

To provide students with practical knowledge of quantitative analysis of materials by classical and instrumental methods for developing experimental skills in building technical competence.

**COURSE OUTCOMES**

On completion of this course, students will be able to:

1. Identify and use different types of instruments for quick and accurate analysis.
2. Apply the various titrimetric analyses for the estimation of metal ions present in raw materials like ores, alloys *etc.*
3. Analyze water sample for hardness, sodium /potassium content, COD and DO.

<b>ENGINEERING CHEMISTRY LABORATORY</b>
---

**Course code: 18CH1ILCHY/18CH2ILCHY**

**L: P: I: S: 0: 2:0:0**

**Exam Hours: 03**

**Credits: 01**

**CIE Marks: 50**

**SEE Marks: 50**

**Course Objectives:**

To provide students with practical knowledge of quantitative analysis of materials by classical and instrumental methods for developing experimental skills in building technical competence.

**Course Outcomes:** On completion of this course, students will be able to,

<b>CO1</b>	Identify and use different types of instruments for quick and accurate analysis.
<b>CO2</b>	Apply the various titrimetric analyses for the estimation of metal ions present in raw materials like ores, alloys <i>etc.</i>
<b>CO3</b>	Analyze water sample for hardness, sodium /potassium content, COD and DO.

**Mapping of course outcomes to program outcomes:**

	<b>PO1</b>	<b>PO2</b>	<b>PO3</b>	<b>PO4</b>	<b>PO5</b>	<b>PO6</b>	<b>PO7</b>	<b>PO8</b>	<b>PO9</b>	<b>PO10</b>	<b>PO11</b>	<b>PO12</b>
<b>CO1</b>	3	2	---	---	---	---	---	---	---	---	---	---
<b>CO2</b>	3	2	---	---	---	---	---	---	---	---	---	---
<b>CO3</b>	3	2	---	---	---	---	---	---	---	---	---	---

### LIST OF EXPERIMENTS

Experiment No.	Course Content	COs
1.	Estimation of copper in industrial waste water effluent by using colorimeter	CO1
2.	Estimation of strength of an acid mixture by conductometry	CO1
3.	Determination of pKa value of a weak acid using pH meter	CO1
4.	Determination of viscosity coefficient of a given lubricant /polymer using Ostwald's viscometer	CO1
5.	Estimation of Iron in mild steel solution using standard $K_2Cr_2O_7$ solution by potentiometry	CO1
6.	Determination of percentage of copper in brass solution by Iodometric method	CO2
7.	Determination of Iron in the given sample of rust solution by external indicator method	CO2
8.	Determination of Calcium Oxide in the given sample of cement by rapid EDTA method	CO2
9.	Assessment of total hardness in a sample of water by complexometric method	CO3
10.	Determination of Chemical Oxygen Demand of the given industrial waste Water sample	CO3
11.	Determination of Dissolved Oxygen in the given water sample by Winkler's method (Demo)	CO3
12.	Estimation of Sodium & Potassium by Flame photometric method in a water sample (Demo)	CO3

#### Reference books:

1. J. Bassett, R.C. Denny, G.H. Jeffery, A. I. Vogel, Text book of quantitative inorganic analysis, 4<sup>th</sup> Edition, 1978.
2. O. P. Vermani & Narula, "Theory and Practice in Applied Chemistry" New Age International Publisher, second edition, 2012.
3. Gary D. Christian, "Analytical chemistry", Wiley India, 6<sup>th</sup> edition, 2007.
4. DSCE laboratory manual 2018-19.

<b>Assessment Pattern</b>		
<b>CIE –Continuous internal evaluation lab (50 Marks)</b>		
<b>Bloom's Category</b>	<b>Performance (Day To Day)</b>	<b>Internal Test</b>
<b>Marks (Out of 50)</b>	<b>25</b>	<b>25</b>
Remember	10	10
Understand	05	05
Apply	05	05
Analyze	05	05
Evaluate	--	--
Create	--	--

<b>Assessment Pattern</b>	
<b>SEE –Semester end Examination lab (50 Marks)</b>	
<b>Bloom's Category</b>	<b>Lab Marks(50)</b>
Remember	10
Understand	05
Apply	20
Analyze	15
Evaluate	--
Create	--

**Assessment Pattern:**

CIE in Laboratory (50 Marks)				
Performance of the experiment	15	25 (A)	Total=A+B+C =50	Total out of 50 marks
Record	10			
Internal test	40	Reduced to 15		
Viva	10	Viva 10(C)		
Total CIE marks for Lab = 50 Marks				
* SEE Lab will be conducted for 50 marks				

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**DAYANANDA SAGAR COLLEGE OF ENGINEERING**  
**DEPARTMENT OF CHEMISTRY**  
**BENGALURU - 560078**

**DO's**

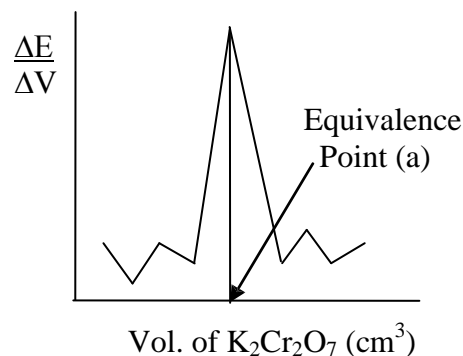
- Maintain discipline when you are in the laboratory.
- Adhere and follow timings, proper dress code with appropriate footwear.
- Secure long hair, loose clothing & know safety and emergency procedures.
- Bags, and other personal items must be stored in designated place.
- Come prepared with viva, procedure, and other details of the experiment.
- Keep water and gas taps closed except when these utilities are needed.
- Inspect all equipment/apparatus for damage prior to use.
- Handle the apparatus/apparatus gently and with care
- Conduct the experiments accurately as directed by the teacher.
- Switch off the power supply after completion of experiment.
- Immediately report any sparks/ accidents/ injuries/ any other untoward incident to the faculty/ instructor.
- In case of an emergency or accident, follow the safety procedure.
- Switch OFF the power supply after completion of experiment

**DONT's**

- The use of mobile/ any other personal electronic gadgets is prohibited in the laboratory.
- Do not make noise in the Laboratory & do not sit on experimental table.
- Do not throw waste such as tissue paper, filter paper, etc., in to the sink.
- Never taste/smell any chemicals unless instructed to do so and do not allow chemicals to come in contact with your skin and eyes.
- Do not pipette out concentrated acids and strong alkalis.
- Do not weigh an object or a sample when it is hot.
- Do not keep alcohol or ether near the flame.
- Do not walk barefooted in the laboratory.
- Do not leave the Laboratory without the signature of the concerned staff in manual book.

**OBSERVATION & CALCULATIONS**

Volume of $K_2Cr_2O_7$ ( $cm^3$ )	E (mV)	$\Delta E$	$\Delta V$	$\frac{\Delta E}{\Delta V}$
0		NIL	NIL	NIL
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				



From the graph, volume of  $K_2Cr_2O_7$  at equivalence point = \_\_\_\_\_  $cm^3$  (a)

Normality of  $K_2Cr_2O_7$  = \_\_\_\_\_ N (will be given) ----- (b)

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

$$N = \frac{b \times a}{25} = \text{_____} \text{ N (z)}$$

Weight of FAS /  $dm^3$  = Normality of FAS  $\times$  Equivalent weight of FAS (392.24)

$$= z \times 392 \text{ g} = \text{_____} \text{ g/litre (w)}$$

$$\text{Weight of FAS in the given solution} = \frac{w \times 25}{1000}$$

**Result:** Weight of FAS in given solution = \_\_\_\_\_ g/litre

## **PART-A**

### **EXPERIMENT-1**

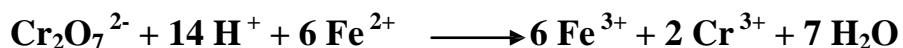
#### **ESTIMATION OF FAS USING STANDARD $K_2Cr_2O_7$ SOLUTION BY POTENTIOMETRY**

##### **Principle:**

The estimation of amount of substance present in a solution by the measurement of emf is known as potentiometric titration. Here, emphasis is laid on the change in emf of cell as a titrant is added. A simple arrangement for potentiometric titration has a reference electrode (saturated calomel electrode) and an indicator electrode (platinum electrode).

The emf of the cell containing the solution is determined by adding increments of the titrant solution until the equivalence point is reached and emf is determined after each addition. After reaching equivalence point, emf increases slightly on the excess addition of titrant.

For the redox Potentiometric titration of FAS against  $K_2Cr_2O_7$  the indicator electrode is a bright platinum electrode and the oxidising agent ( $K_2Cr_2O_7$ ) is placed in the burette. The reaction proceeds as follows.



##### **Procedure:**

Take 25 cm<sup>3</sup> of the given FAS solution into a clean beaker and add 2 test tubes of dil.  $H_2SO_4$ . Immerse platinum electrode and saturated calomel electrode assembly, connect to a potentiometer and note down the potential value in milli volts (mV). Fill a micro burette with the given standard  $K_2Cr_2O_7$  solution.

Add  $K_2Cr_2O_7$  solution in increments of 0.5 cm<sup>3</sup> to FAS solution in the beaker. Stir the solution thoroughly and measure the emf after each addition. When the reaction reaches the equivalence point, there is a maximum increase in the emf value. After the equivalence point, there will be slight increase in emf on continued addition of  $K_2Cr_2O_7$ .

Graph of  $\Delta E/\Delta V$  against volume of  $K_2Cr_2O_7$  is plotted. From the graph, volume of  $K_2Cr_2O_7$  at equivalence point is found out.

**OBSERVATION & CALCULATIONS**

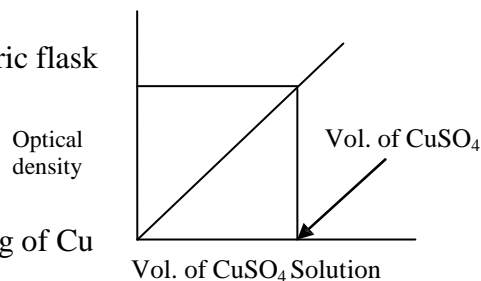
Flask No	Volume of $\text{NH}_3$ ( $\text{cm}^3$ )	Volume of copper sulphate solution ( $\text{cm}^3$ )	Absorbance (Optical Density)
Blank (B)	5	0	
1	5	5	
2	5	10	
3	5	15	
4	5	20	
5	5	25	
TS	5	Test solution	

Molecular Weight of  $\text{CuSO}_4 = 249.54 \text{ g}$

2g of  $\text{CuSO}_4$  dissolved in  $250 \text{ cm}^3$  of water in a volumetric flask

249.54 g of  $\text{CuSO}_4$  contains 63.5 g of Cu

$$\text{Therefore, 2 g. of } \text{CuSO}_4 = \frac{63.54 \times 2}{249.55} = 0.5092 \text{ g of Cu}$$



$250 \text{ cm}^3$  of  $\text{CuSO}_4$  contains = 0.5092 g of Cu

i.e.,  $1 \text{ cm}^3$  of  $\text{CuSO}_4$  contains =  $\frac{0.5092}{250} = 2.03 \times 10^{-3} \text{ g of Cu}$

Volume of test solution from the graph ('Z'  $\text{cm}^3$ ) = ..... $\text{cm}^3$

Therefore, weight of copper in the given test solution =  $2.03 \times 10^{-3} \times \text{'Z'} = \dots\dots\dots\text{g}$

**Result:** The weight of the copper in water effluent = .....g

## EXPERIMENT-2

### ESTIMATION OF COPPER IN INDUSTRIAL WASTE WATER EFFLUENT BY USING COLORIMETER

#### Principle:

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

$$I_0 = I_a + I_r + I_t$$

For a glass- air interface,  $I_r$  is negligible. Therefore,  $I_0 = I_a + I_t$ .  $I_t/I_0 = T$  called the transmittance,  $\log 1/T = \log I_0/I_t$  is called the absorbance or optical density  $A$ . The relation between absorbance  $A$ , concentration  $C$  (Expressed in mol/liter) and path length  $t$  (expressed in cm) is given by Beer Lambert's law.

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

Where  $C$  is the concentration,  $t$  is the path length.  $\epsilon$  is a molar extinction coefficient for a given solution at a given wavelength. If path length ( $t$ ) is kept constant, then  $A \propto C$ . Hence plot of absorbance against concentration gives a straight line. A series of standard solutions of copper is treated with ammonia to get blue **cuprammonium** complex 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 volume of copper sulphate ( $\text{cm}^3$ ) to get a calibration curve. A known volume of the test solution is treated with strong ammonia and diluted as above. The absorbance of this solution at 620 nm is measured and its concentration is determined from the calibration curve.

#### Procedure:

Transfer the given copper sulphate solution (stock solution) to a burette and draw out 5, 10, 15, 20 and 25  $\text{cm}^3$  of the solution into 50  $\text{cm}^3$  volumetric flasks. Add 5  $\text{cm}^3$  of ammonia solution to each of them and dilute up to the mark with ion exchange water. Stopper the flasks and mix. To the test solution given in a 50  $\text{cm}^3$  volumetric flask, add 5  $\text{cm}^3$  of ammonia and dilute upto the mark and mix well. Prepare a blank solution by diluting 5  $\text{cm}^3$  of ammonia solution in a 50  $\text{cm}^3$  volumetric flask up to the mark with ion exchange water and mix well. Measure the absorbance of the solutions against blank at 620 nm using photoelectric colorimeter. Draw a calibration curve by plotting optical density (OD) against volume of copper sulphate ( $\text{cm}^3$ ). Using the calibration curve, find out the concentration of copper in the test solution and calculate the amount of copper in 50  $\text{cm}^3$  of the given test solution.

**OBSERVATION & CALCULATIONS**

Sl. No	Volume of NaOH added (cm <sup>3</sup> )	Conductivity (Ohm <sup>-1</sup> cm <sup>-1</sup> )	Sl. No	Volume of NaOH added (cm <sup>3</sup> )	Conductivity (Ohm <sup>-1</sup> cm <sup>-1</sup> )	Sl. No	Volume of NaOH added (cm <sup>3</sup> )	Conductivity (Ohm <sup>-1</sup> cm <sup>-1</sup> )
1.	00		13.	6.0		25.	12.0	
2.	0.5		14.	6.5		26.	12.5	
3.	1.0		15.	7.0		27.	13.0	
4.	1.5		16.	7.5		28.	13.5	
5.	2.0		17.	8.0		29.	14.0	
6.	2.5		18.	8.5		30.	14.5	
7.	3.0		19.	9.0		31.	15.0	
8.	3.5		20.	9.5		32.	15.5	
9.	4.0		21.	10.0		33.	16.0	
10.	4.5		22.	10.5		34.	17.5	
11.	5.0		23.	11.0		35.	18.0	
12.	5.5		24.	11.5		36.	18.5	

Normality of NaOH = 1.0 N

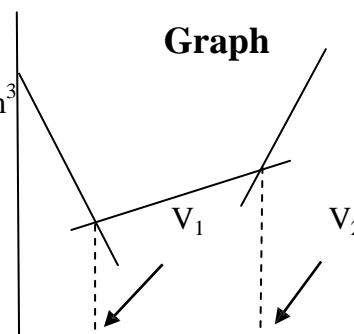
Volume of acid mixture taken = 50 cm<sup>3</sup>

Volume of NaOH required to neutralize HCl = V<sub>1</sub> = \_\_\_\_\_ cm<sup>3</sup>

Volume of NaOH required to neutralize acid mixture V<sub>2</sub> = \_\_\_\_\_ cm<sup>3</sup>

Volume of NaOH required to neutralize CH<sub>3</sub>COOH (V<sub>2</sub> - V<sub>1</sub>) = \_\_\_\_\_ cm<sup>3</sup>

Specific Conductance (ohm<sup>-1</sup> cm<sup>-1</sup>)



Normality of HCl =  $\frac{(N \times V_1)_{\text{NaOH}}}{\text{Vol. of acid mixture}}$  = \_\_\_\_\_ = ..... N (a)

Weight of HCl present in 1dm<sup>3</sup> of the acid mixture = N<sub>HCl</sub> × Eq. wt. of HCl = a × 36.46 = \_\_\_\_\_ g/litre

Normality of CH<sub>3</sub>COOH =  $\frac{N_{\text{NaOH}} \times (V_2 - V_1)}{\text{Volume of acid mixture}}$  = \_\_\_\_\_ N (b)

Weight of CH<sub>3</sub>COOH/dm<sup>3</sup> = N<sub>CH<sub>3</sub>COOH</sub> × Eq. weight of CH<sub>3</sub>COOH = b × 60.05 = \_\_\_\_\_ g/ litre

**Result:**

Weight of HCl present in the given acid mixture = \_\_\_\_\_ g/ litre

Weight of CH<sub>3</sub>COOH in the given acid mixture = \_\_\_\_\_ g/ litre

### EXPERIMENT-3

## ESTIMATION OF STRENGTH OF AN ACID MIXTURE BY CONDUCTOMETRY

#### Principle:

The neutralization point of acid solution in acid – base titration is determined by a change in the colour of the indicator. Such a determination can be carried out by conductometric titration. In a conductometric titration a change in conductance of the solution near the neutralization point takes place. However the change is not sharp and hence the neutralization point is determined graphically by plotting conductivity against titre values. The principle underlying conductometric titration is the replacement of ions of a particular conductivity by ions of different conductivity during titration.

Conductometric titration may be applied for the determination of concentration of acids present in a mixture. In the titration of a mixture of a weak acid ( $\text{CH}_3\text{COOH}$ ) and a strong acid ( $\text{HCl}$ ) against a strong base ( $\text{NaOH}$ ), the conductance initially decreases upon adding  $\text{NaOH}$  to acid mixture owing to the substitution of highly mobile  $\text{H}^+$  ions (mobility:  $350 \text{ Ohm}^{-1} \text{ cm}^{-1}$ ) by the less mobile  $\text{Na}^+$  ion (mobility:  $50 \text{ ohm}^{-1} \text{ m}^{-1}$ ) and this trend continues till all the  $\text{H}^+$  ions of  $\text{HCl}$  are replaced i.e, the strong acid get neutralized. Later, conductance slowly increases as  $\text{CH}_3\text{COOH}$  is converted into its salt,  $\text{CH}_3\text{COONa}$ . Further addition of  $\text{NaOH}$  raises the conductance steeply due to the presence of free  $\text{Na}^+$  and  $\text{OH}^-$  ions (mobility  $198 \text{ ohm}^{-1} \text{ m}^{-1}$ ).

#### Procedure:

Fill a micro burette with the standard  $\text{NaOH}$  solution. Pipette out  $50 \text{ cm}^3$  of the given acid mixture into clean  $100 \text{ cm}^3$  beaker. Place the conductivity cell in the beaker and connect cell to conductometer and measure the conductance. Add  $0.5 \text{ cm}^3$   $\text{NaOH}$  to beaker and measure conductance. With the addition of  $\text{NaOH}$  (with increments of  $0.5 \text{ cm}^3$ ), initially conductance decreases and then rises slowly and finally rises sharply. Continue titration until the conductance is almost same as it was in the beginning of the titration. Plot a graph of conductance (Y- axis) versus volume of  $\text{NaOH}$  (X- axis). The point  $V_1$  corresponds to neutralization of  $\text{HCl}$  and  $V_2$  corresponds to neutralization of both hydrochloric and acetic acids. The difference in the volumes corresponding to the points  $V_2$  and  $V_1$  gives the volume required to neutralize  $\text{CH}_3\text{COOH}$ .

**OBSERVATION AND CALCULATIONS****DETERMINATION OF SODIUM:**

Weight of Sodium per ml of the solution = 1 mg

1ml of NaCl solution contains 0.002542g of NaCl

58.5 g of NaCl contains 23 g of Na

$$0.002542 \text{ g of NaCl contains } = \frac{23}{58.5} \times 0.002542$$

$$= 1 \text{ mg}$$

Therefore, 1ml of NaCl solution contains 1 mg of Na

Vol. of NaCl pipette out in ml	Concentration of Na (in mg)	Emission Response
5		
10		
15		
20		
25		
Test solution		

1ml of NaCl solution contains 0.002542g of NaCl

Therefore X ml of NaCl solution contains =

$$X \times 0.002542 \text{ g of NaCl} = \text{-----} \times 0.002542 \text{ g of NaCl}$$

$$= \text{-----g of NaCl (Y)}$$

Therefore the amount of Na present in above test solution (X ml) can be calculated by knowing the equivalent weight of Na and molecular weight of NaCl.

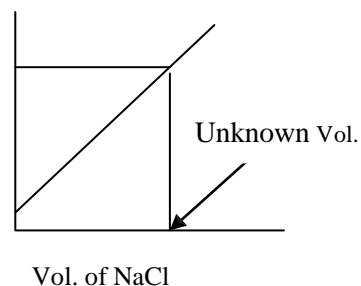
Therefore, Y g of NaCl contains

$$23$$

$$= \text{-----} \times Y = \text{-----g} = \text{-----mg}$$

$$58.5$$

Flame photometer reading of Na

**DETERMINATION OF POTASSIUM:**

Weight of potassium per ml of the solution = 1 mg

1ml of KCl solution contains (0.001909g of KCl)

74.5 g of KCl contains 39 g of K

39

$$\text{-----} \times 0.001909 = 1 \text{ mg}$$

74.5

Therefore, 1ml of KCl solution contains 1 mg of K

Vol of KCl pipette out in ml	Concentration of K (in mg)	Emission Response
5		
10		
15		
20		
25		
Test solution		

1ml of KCl solution contains 0.001909g of KCl

Therefore, X ml of KCl solution contains = X ×

$$0.001909 \text{ g of KCl} = \text{-----} \times 0.001909 \text{ g of KCl}$$

$$= \text{-----g of KCl (Y)}$$

Therefore, the amount of K present in above test solution (X ml) can be calculated by knowing the equivalent weight of K and molecular weight of KCl

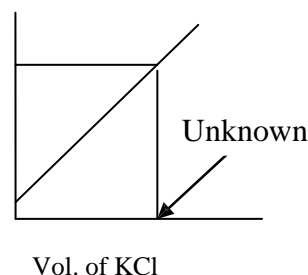
$$39$$

$$\text{Therefore, Y g of KCl contains } = \text{-----} \times Y = \text{-----g}$$

$$74.5$$

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

Flame photometer reading of K



**Result:** The weight of Na<sup>+</sup> present in the given test solution = -----mg

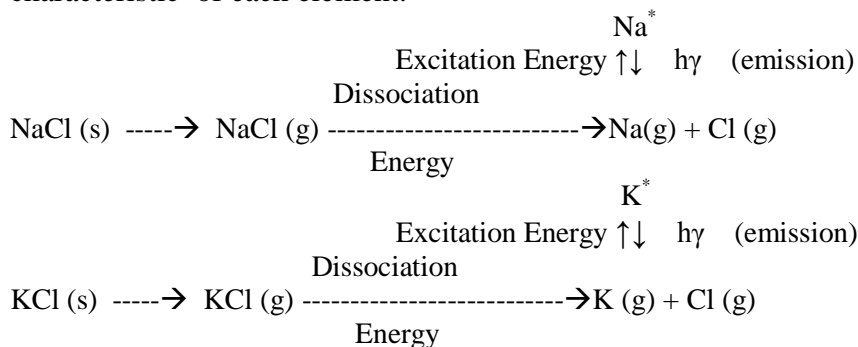
The weight of K<sup>+</sup> present in the given test solution = -----mg



## EXPERIMENT-4

### ESTIMATION OF SODIUM AND POTASSIUM BY FLAME PHOTOMETRIC METHOD IN A WATER SAMPLE

**Principle:** Flame photometry is an atomic emission technique used for the detection of metals. If a solution containing metallic salts is aspirated into a flame, a vapour, which contains metallic atoms, will be formed. The electrons from the metallic atoms are then excited from ground state ( $E_1$ ) to higher energy state ( $E_n$ ) where  $n = 2, 3, 4, \dots, 7$ , by making use of thermal energy of flame. From higher energy states, these electrons will return to the ground state by emitting radiations ( $E_n - E_1 = h\nu$  where  $n = 2, 3, 4, \dots, 7$ ) which are the characteristic of each element.



Flame photometer correlates the emitted radiations with the concentration of these elements. It is simple and rapid method for the elements that can be easily excited (sodium and other alkali metals). A flame photometer is composed of the pressure regulator and flow meter for fuel gases, an atomiser, burner, photosensitive detector and output recorder. A filter of the element whose concentration is to be determined is inserted between the flame and the detector. Propane gas is used as fuel and air or oxygen is used as oxidant. Combination of these two will give a temperature of  $1900^\circ\text{C}$ . The whole analysis depends on the flow rate of the fuel, oxidant, the rate of introduction of the sample and droplet size.

The sample containing the analyte is aspirated into the flame through atomiser. Radiation from resulting flame is collected by the lens and allowed to pass through an optical filter, which permits only the radiation characteristic of the element under investigation into the photocell. The output from the photocell represents the concentration and nature of the element.

#### Procedure for determination of Sodium and Potassium:

##### Determination of Sodium:

Transfer 5, 10, 15, 20 and 25  $\text{cm}^3$  of standard sodium chloride solution (which is prepared by weighing accurately 2.542g NaCl into a 1 liter volumetric flask and dissolving the crystals and diluting the solution upto the mark with distilled water and mixing. The solution gives 1 ppm /ml) into 100 ml standard volumetric flasks and dilute upto the mark with distilled water. Place the distilled water in the suction capillary of the instrument and set the instrument to read zero. Place each of the standard solutions in the suction capillary and set the instrument to read 5, 10, 15, 20 and 25 respectively (rinse with distilled water between each reading).

Dilute the given test solution upto the mark, shake well and place the solution in the suction capillary and record the reading. Draw a calibration curve by plotting the reading (y-axis) and volume of NaCl solution (x-axis). From the calibration curve, find out the volume of the given test solution and from which calculate the amount of Na (58.5 g of NaCl contains 23 g of Na).

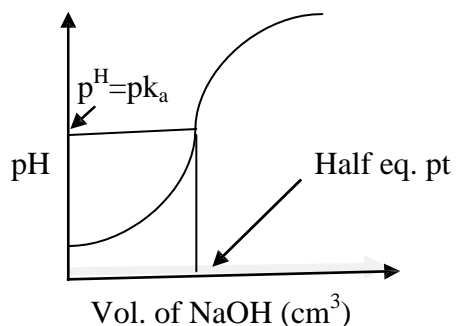
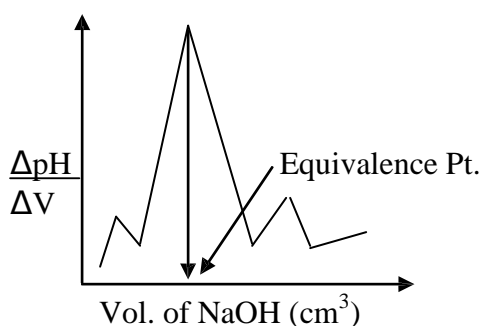
**Determination of Potassium:** Prepare standard solution of potassium and follow the same procedure given above for sodium.

**OBSERVATION & CALCULATIONS**

Volume of NaOH (cm <sup>3</sup> )	p <sup>H</sup>	$\Delta p^H$	$\Delta V$	$\frac{\Delta p^H}{\Delta V}$	Volume of NaOH (cm <sup>3</sup> )	p <sup>H</sup>	$\Delta p^H$	$\Delta V$	$\frac{\Delta p^H}{\Delta V}$
0.0		NIL	NIL	NIL	5.5				
0.5					6.0				
1.0					6.5				
1.5					7.0				
2.0					7.5				
2.5					8.0				
3.0					8.5				
3.5					9.0				
4.0					9.5				
4.5					10.0				
5.0					10.5				

Nature of Graph:

Derivative Curve

At the half equivalence point,  $p^H = pK_a$ **Result:** pKa of the given weak acid = \_\_\_\_\_

## EXPERIMENT-5

### DETERMINATION OF $pK_a$ VALUE OF A WEAK ACID USING pH METER

#### Principle:

During the titration of an acid with a base, the pH of the solution rises gradually at first and then more rapidly until at the equivalence point, there is a very sharp increase in pH for a very small quantity of added base. After the equivalence point, the pH increases only slightly on addition of excess base.

According to Henderson Hasselbalch equation,

$$p^H = pK_a + \log [\text{salt}] / [\text{acid}]$$

Where  $K_a$  is dissociation constant of the acid and  $pK_a = -\log K_a$ . At half equivalence point,  $[\text{salt}] = [\text{acid}]$  and hence  $pH = pK_a$ . Therefore, pH at half equivalence point gives the  $pK_a$  of weak acid.

#### Procedure:

Take 50 cm<sup>3</sup> of the given weak acid (acetic acid or formic acid etc) into a beaker. Immerse a glass electrode – calomel electrode assembly into the acid and connect to a pH meter and measure pH of the acid. Blow the solution thoroughly using a glass tube and measure the  $p^H$  after each addition of NaOH in the increments of 0.5 cm<sup>3</sup>. pH increases gradually in the beginning and near the equivalence point there is considerable increase in pH, after the equivalence point there will be only slight increase in pH on further addition of NaOH.

Plot graph of  $\Delta pH / \Delta V$  versus volume of NaOH added and find out equivalence point. Plot another graph of pH against volume of NaOH added and determine pH at half equivalence point. This is  $pK_a$  value of given weak acid.

**OBSERVATION & CALCULATIONS**

	<b>Trial I</b>	<b>Trial II</b>	<b>Trial III</b>	<b>Average</b> (in seconds)
<b>Test liquid (t<sub>l</sub>)</b> (in seconds)				
<b>Water (t<sub>w</sub>)</b> (in seconds)				

Lab temperature = ..... °C

$\eta_w$  = Viscosity coefficient of water = ..... millipoise

$d_w$  = Density of water = ..... g/cm<sup>3</sup>

$d_l$  = Density of given liquid = ..... g/cm<sup>3</sup>

Viscosity coefficient of given liquid ( $\eta_l$ ) =  $\frac{t_l d_l}{t_w d_w} \times \eta_w$

= \_\_\_\_\_

= \_\_\_\_\_ milli poise

**Result:** Viscosity coefficient of the given organic liquid = \_\_\_\_\_ milli poise

**EXPERIMENT-6****DETERMINATION OF VISCOSITY CO-EFFICIENT OF A GIVEN LUBRICANT/POLYMER USING OSTWALD'S VISCOMETER****Principle:**

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,

$$\eta_l = \frac{\pi p r^4 t}{8 v l}$$

Where  $v$  = volume of the liquid,  $r$  = radius of the tube,  $l$  = length of the tube,  $p$  is the pressure between the two ends of the tube,  $\eta$  is the coefficient of viscosity of the liquid. If equal volume of two different liquids are allowed to flow through the same tube with identical conditions then,

$$\frac{\eta_l}{\eta_w} = \frac{t_l d_l}{t_w d_w} \quad (l - \text{Liquid}, w - \text{Water})$$

The time  $t_1$  taken by the given liquid to travel through a certain distance in the tube is determined. The time  $t_2$  taken by a standard liquid (water) to travel through the same distance is measured. Knowing the densities  $d_1$  and  $d_2$  of the test liquid and the standard (water) and the viscosity coefficient of the standard, coefficient of viscosity of the test liquid can be calculated.

**Procedure:**

Take clean and dried viscometer and fix it vertically to a stand. Then transfer 20 cm<sup>3</sup> (using pipette) of the test liquid into the wider limb of the viscometer. Suck it above the upper mark of the viscometer and allow it to flow freely through the capillary. When the level of liquid just crosses the upper mark, start a stop clock and stop the stop clock when it just crosses the lower mark, and determine the time  $t_1$  taken in seconds by the liquid to flow between fixed points. Repeat to get agreeing values. Pour the liquid and wash with water, rinse with acetone and dry it. Fix the viscometer vertically to a stand and take 20 cm<sup>3</sup> of the water and suck it above the upper mark, allow it to flow freely through the capillary. When the level of water just crosses the upper mark, start a stop clock and stop it when the water just crosses the lower mark. Note the time of flow in seconds ( $t_w$ ). Repeat to get agreeing values. By using  $t_l$  and  $t_w$  values, determine the viscosity coefficient of given liquid.

**OBSERVATION & CALCULATIONS**

Weight of weighing bottle + EDTA salt = .....g

Weight of empty weighing bottle = .....g

Weight of EDTA salt = .....g

Molarity of EDTA Solution =  $\frac{\text{Weight of EDTA} \times 4}{\text{Molecular weight of EDTA} \times 1000} = \frac{\text{.....} \times 4}{372.24 \times 1000} = \text{..... M}$

Burette readings	Trial I	Trial II	Trial III
Final burette reading (cm <sup>3</sup> )			
Initial burette reading (cm <sup>3</sup> )			
Volume of EDTA solution run down in (cm <sup>3</sup> )			

Volume of EDTA consumed = .....cm<sup>3</sup> $(M \times V)_{\text{Hard water}} = (M \times V)_{\text{EDTA}}$ 

$$M_{\text{Hard water}} = \frac{(M \times V)_{\text{EDTA}}}{25} = \frac{\text{.....}}{25} = \text{..... M}$$

Weight per liter of CaCO<sub>3</sub> equivalent hardness =  $M_{\text{Hard water}} \times \text{Mol. Wt. of CaCO}_3 (100)$   
 = .....  $\times 100$  = ..... g/liter (Z)

Hardness of water sample =  $\frac{Z \times 10^6}{1000} = \frac{\text{.....} \times 10^6}{1000}$  mg/litre of CaCO<sub>3</sub> = .....mg/liter of CaCO<sub>3</sub>

**Result:** The total hardness of the given water sample = ..... mg/litre or ppm of CaCO<sub>3</sub>

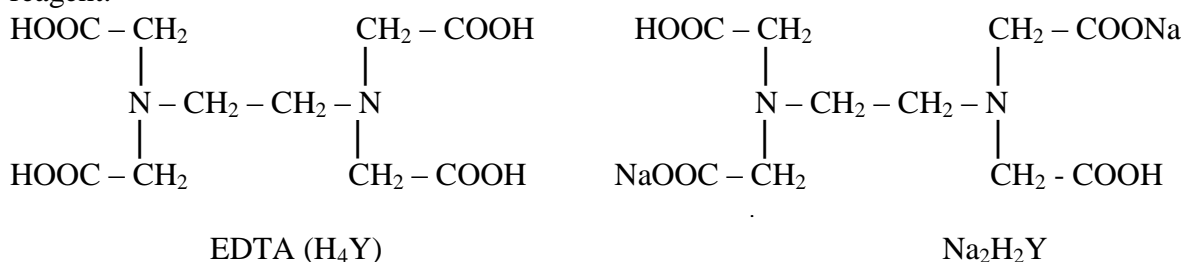
## **PART-B**

### **EXPERIMENT-1**

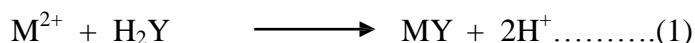
#### **ASSESSMENT OF TOTAL HARDNESS IN A SAMPLE OF WATER BY COMPLEXOMETRIC METHOD**

**Principle:**

Hardness of water is due to the presence of calcium and magnesium salts in water. Ethylene diamine tetra acetic acid (EDTA) forms complexes with a large number of cations including  $\text{Ca}^{++}$  and  $\text{Mg}^{++}$  ions. So it is possible to determine the total hardness of water using EDTA reagent.



The disodium salt of EDTA molecule ( $\text{H}_2\text{Y}$ ) has two easily replaceable hydrogen atoms and after ionization it is represented as  $\text{H}_2\text{Y}^{2-}$ . The later forms complexes with metal ions as follows.



where  $\text{M}^{2+}$  is  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  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  $\text{H}^+$  ions, buffer mixture has to be used to maintain a pH of 10. The buffer mixture used in the titration is  $\text{NH}_3 - \text{NH}_4\text{Cl}$ . The hardness of water is usually expressed in terms of ppm (parts per million) of  $\text{CaCO}_3$ . Since EDTA (free acid) is sparingly soluble, its disodium salt,  $\text{Na}_2\text{H}_2\text{Y}$  is used for preparing the reagent.

**Procedure:**

**Part A- Preparation of standard solution of disodium salt of EDTA** – Weigh accurately the given disodium salt of EDTA crystals using an electronic weighing balance. Note the weight, transfer the crystals carefully into a funnel placed over a 250 cm<sup>3</sup> volumetric flask and note down the empty weight of the weighing bottle. Add small amount of water allowing all the crystals to run down into the flask and then ½ test tube of ammonia. 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. Calculate the molarity of EDTA.

**Part B - Determination of total hardness of water sample** - Pipette out 25 cm<sup>3</sup> of the given water sample into a clean conical flask. Add 5 cm<sup>3</sup> of  $\text{NH}_3 - \text{NH}_4\text{Cl}$  buffer and a pinch of Eriochrome Black – T indicator. Titrate against standard EDTA solution till the colour of the solution changes from wine red to clear blue. Repeat the titration for concordant values.

**OBSERVATION & CALCULATIONS**

Weight of weighing bottle + EDTA salt = .....g

Weight of empty weighing bottle = .....g

Weight of EDTA salt = .....g

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

Burette : Std. EDTA solution

Conical flask : 25 cm<sup>3</sup> of cement solution + 5ml glycerol + 5ml diethyl amine + 4 N NaOH

Indicator : Patton and Reeder's

End point : Wine red to clear blue

Burette readings	Trial I	Trial II	Trial III
Final burette reading (cm <sup>3</sup> )			
Initial burette reading (cm <sup>3</sup> )			
Volume of EDTA solution consumed (cm <sup>3</sup> )			

Volume of EDTA consumed = .....cm<sup>3</sup>

$$(M \times V)_{\text{Cement Solution}} = (M \times V)_{\text{EDTA}}$$

$$M_{\text{Cement solution}} = \frac{(M \times V)_{\text{EDTA}}}{V_{\text{cement solution}}} = \frac{\quad \times \quad}{25} = \quad \text{M}$$

$$\text{Weight of CaO present in 250ml} = \frac{M_{\text{Cement solution}} \times \text{Mol. Wt. of CaO (56)}}{4} \times \frac{56}{4} = \quad \text{(a)}$$

$$\text{Weight of Cement} = \quad \text{g (W)}$$

$$\text{Percentage of CaO in the given cement solution} = \frac{a}{W} \times 100 = \quad$$

**Result:** The amount of CaO present in the given cement solution = \_\_\_\_\_ %



**EXPERIMENT-2****DETERMINATION OF CALCIUM OXIDE IN THE GIVEN SAMPLE OF CEMENT BY RAPID EDTA METHOD****Principle:**

Cement contains silicates of iron, aluminum and calcium. Calcium is a prime constituent of cement. The general composition of a Portland cement is given here.

Constituent	Percentage
CaO	60.0-66.0
SiO <sub>2</sub>	17.0-25.0
Al <sub>2</sub> O <sub>3</sub>	3.0-8.0
Fe <sub>2</sub> O <sub>3</sub>	2.0-6.0
MgO	0.1-5.5
Na <sub>2</sub> O & K <sub>2</sub> O	0.5-1.5
SO <sub>3</sub>	1.0-3.0

In the estimation of calcium in cement, the given cement is treated with concentrated hydrochloric acid. The insoluble silica residue is removed by filtration. Iron and aluminium in the filtrate are converted as their hydroxides and filtered. Filtrate containing calcium ions is titrated at a pH of 12-14 against EDTA using Patton and Reeder's indicator. The latter is used because, Eriochrome Blank-T forms very weak complex with calcium ions. Diethylamine is added to maintain a pH about 12 and glycerol is added to get a sharp end point. This method facilitates the determination of calcium only, although Mg<sup>2+</sup> ions are present in the cement solution which can be suppressed by adding NaOH.

**Procedure:****Part A- Preparation of standard solution of disodium salt of EDTA**

Weigh accurately the given disodium salt of EDTA crystals using an electronic weighing balance. Note the weight, transfer the crystals carefully into a funnel placed over a 250 cm<sup>3</sup> volumetric flask and note down the empty weight of the weighing bottle. Add little water and 1/2 test tube of ammonia. Dissolve the crystals by swirling the flask gently. Add some more water if needed. Dilute the solution upto 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. Calculate the molarity of EDTA.

**Part B: Estimation of CaO**

Pipette out 25 cm<sup>3</sup> of the given cement solution into a clean conical flask. Add 2 cm<sup>3</sup> of 1:1 glycerol with constant shaking of the contents of the flask, add 2 cm<sup>3</sup> of diethyl amine and 1/2 test tube of 4N NaOH. Add 2 drops of Patton and Reeder's indicator. Titrate against the EDTA solution till the colour changes sharply from wine red to clear blue. Repeat the titration for concordant values.

**OBSERVATION & CALCULATIONS**

Weight of brass sample = \_\_\_\_\_ g

Burette readings	Trial I	Trial II	Trial III
Final burette reading (cm <sup>3</sup> )			
Initial burette reading (cm <sup>3</sup> )			
Volume of sodium thiosulphate (cm <sup>3</sup> )			

**Calculations:**

$$(N \times V)_{\text{Brass Solution}} = (N \times V)_{\text{Na}_2\text{S}_2\text{O}_3}$$

$$N_{\text{Brass Solution}} = \frac{(N \times V)_{\text{Na}_2\text{S}_2\text{O}_3}}{V_{\text{Brass Solution}}} = \text{_____} N \text{ (a)}$$

$$\begin{aligned} \text{Weight of Cu in the given 250 ml of brass solution} &= \frac{(a) \times \text{Eq. wt. of Cu (63.5)}}{4} \\ &= \frac{\text{_____} \times 63.5}{4} = \text{_____} \text{ g (b)} \end{aligned}$$

$$\begin{aligned} \text{Therefore, the percentage of copper in the given} \\ \text{brass sample} &= \frac{\text{.....b.....g} \times 100}{\text{weight of brass sample}} \\ &= \text{_____} \times 100 = \text{_____} \% \end{aligned}$$

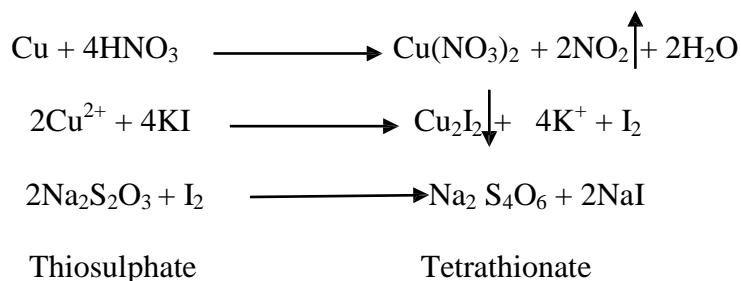
**Result:** The amount of copper present in the given brass sample = .....%

**EXPERIMENT-3****DETERMINATION OF PERCENTAGE OF COPPER IN BRASS  
SOLUTION BY IODOMETRIC METHOD****Principle:**

The chief constituents of brass alloy are **copper and zinc**. It also contains small quantities of tin and iron. The percentage composition of a typical brass alloy is

Cu; 50-90%, Zn; 20-40%, Sn; 0-06%, Pb; 0-02%, Fe; 0-01%

A solution of brass is made by dissolving the sample in minimum amount of nitric acid. Excess of nitric acid is destroyed by boiling with urea. The solution is neutralized and made faintly acidic. Potassium iodide solution is added where the cupric ions oxidize iodide to iodine. The iodine liberated is titrated against sodium thiosulphate. The amount of sodium thiosulphate consumed is a measure of the amount of copper present.

**Procedure:****Part A: Preparation of brass solution**

Dilute the given brass solution in a 250 ml std. flask, add distilled water up to the mark and mix well.

**Part B: Estimation of Copper**

Pipette out 25 cm<sup>3</sup> of above solution into a 250 cm<sup>3</sup> conical flask, add dilute ammonium hydroxide drop wise until a slight blue precipitate is formed. Dissolve the precipitate by adding dilute acetic acid drop wise. Add 1 test tube of 10% KI. Titrate the iodine liberated against standard sodium thiosulphate solution till the mixture turns straw yellow. Add freshly prepared starch indicator and continue the titration till the blue color disappears and white precipitate is left behind. Repeat the titration till the concordant values are obtained.

**OBSERVATION & CALCULATIONS****Part-A: Preparation of Std. solution of  $K_2Cr_2O_7$  solution**Weight of bottle +  $K_2Cr_2O_7$  crystals =  $W_1$  = \_\_\_\_\_ gWeight of empty bottle =  $W_2$  = \_\_\_\_\_ gWeight of  $K_2Cr_2O_7$  crystals transferred =  $(W_1 - W_2)$  = \_\_\_\_\_ g

$$\text{Normality of } K_2Cr_2O_7 \text{ solution} = \frac{\text{Mass of } K_2Cr_2O_7 \times 4}{\text{Eq. weight of } K_2Cr_2O_7 (49)}$$

$$= \frac{\quad \times 4}{49}$$

$$= \quad \text{N}$$

**Part- B: Estimation of iron**

Burette readings	Trial I	Trial II	Trial III
Final burette reading (cm <sup>3</sup> )			
Initial burette reading (cm <sup>3</sup> )			
Volume of potassium dichromate (cm <sup>3</sup> )			

Concordant burette reading = \_\_\_\_\_ cm<sup>3</sup>

$$(N \times V)_{\text{Fe Solution}} = (N \times V)_{K_2Cr_2O_7}$$

$$N_{\text{Fe solution}} = \frac{(N \times V)_{K_2Cr_2O_7}}{V_{\text{Fe solution}}} \times \frac{25}{\quad} = \quad \text{N}$$

$$\text{Weight of Fe in 250 ml of haematite ore solution} = \frac{N_{\text{Fe solution}} \times \text{Eq. wt. of iron (55.85)}}{4}$$

$$= \frac{\quad \times 55.85}{4} = \quad \text{g (a)}$$

Weight of ore taken = 0.98 g (W)

$$\text{Percentage of Iron in the given Haematite ore} = \frac{a}{W} \times 100 = \frac{\quad \times 100}{0.98} = \quad \%$$

**Result:** The percentage of iron in given rust solution = \_\_\_\_\_ %

## EXPERIMENT-4

### DETERMINATION OF IRON IN THE GIVEN SAMPLE OF RUST SOLUTION BY EXTERNAL INDICATOR METHOD

**Principle:**

Haematite is an important ore of iron containing mainly  $\text{Fe}_2\text{O}_3$  and small amount of silica ( $\text{SiO}_2$ ). Iron is present in the solution as ferric ( $\text{Fe}^{3+}$ ) ions in  $\text{Fe}_2\text{O}_3$ . It is reduced to ferrous ( $\text{Fe}^{2+}$ ) ions using stannous chloride ( $\text{SnCl}_2$ ) in hot condition in presence of con.  $\text{HCl}$ . The excess of stannous chloride is oxidized by treating the solution with mercuric chloride. The ferrous ions in the resulting solution titrated against std.  $\text{K}_2\text{Cr}_2\text{O}_7$  using potassium ferricyanide as external indicator. Potassium ferricyanide produces an intense deep blue colour with ferrous ions due to the formation of ferri ferrocyanide complex.

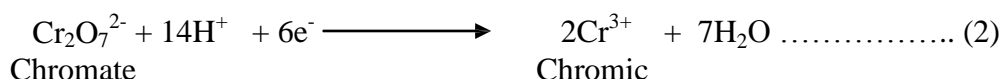
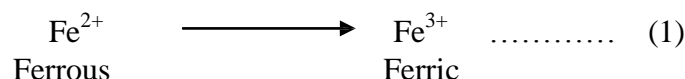
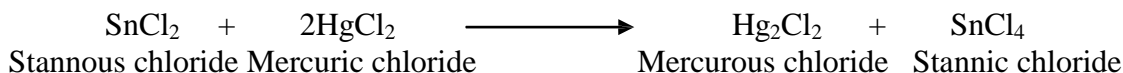
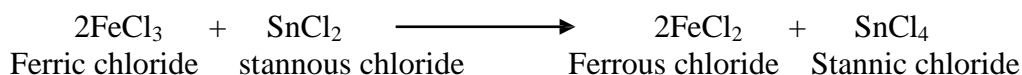
**Procedure:**

**Part A: Preparation of standard potassium dichromate solution** - Weigh out accurately about 1.25 g of  $\text{K}_2\text{Cr}_2\text{O}_7$  crystals and transfer to 250  $\text{cm}^3$  of volumetric flask. Dissolve it in ion exchange water and dilute upto the mark, mix well.

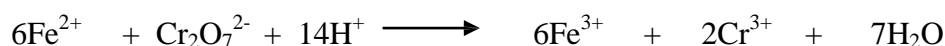
**Part B: Estimation of iron** - Pipette out 25  $\text{cm}^3$  of haematite ore solution into a clean conical flask. Add quarter test tube of conc.  $\text{HCl}$  and heat the solution to boiling. Add stannous chloride to the hot solution drop wise till the yellow solution turns colorless. Add 2 more drops to ensure complete reduction. Cool and add 1 t.t of water and  $\frac{1}{4}$  test tube of mercuric chloride.

Place a number of drops of freshly prepared Potassium ferri cyanide indicator on a wax paper. Add a small quantity of  $\text{K}_2\text{Cr}_2\text{O}_7$  from the burette to the conical flask containing haematite ore solution and mix well. Remove a drop of solution from the conical flask and bring it in contact with a drop of the indicator on the wax paper. The color of the indicator turns blue.

The indicator turns blue as long as the titration is incomplete. Continue the titration by adding  $\text{K}_2\text{Cr}_2\text{O}_7$  in increments of 1  $\text{cm}^3$  at a time and testing as above till a drop of the mixture fails to produce a blue color with the indicator drop on the wax paper (Clean the glass rod after every test). Repeat the titration by taking another 25  $\text{cm}^3$  of the haematite ore solution.



Adding (1) & (2)



**OBSERVATION & CALCULATIONS****Part A: Preparation of standard Ferrous Ammonium Sulphate (Mohr's Salt) solution**

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

$$\text{Normality of FAS solution} = \frac{\text{Weight of FAS} \times 4}{\text{Equivalent weight of FAS}} \times 4 = \frac{\quad \times 4}{392} = \text{-----N (a)}$$

**Part B: Determination of COD of waste water sample.****Back Titration:**

- Burette : Standard FAS solution  
 Conical flask : 25 cm<sup>3</sup> of waste water sample + 10cm<sup>3</sup> of standard Potassium dichromate Solution + 1 t t of 1:1 H<sub>2</sub>SO<sub>4</sub>  
 Indicator : 2-3 drops of ferroin  
 End point : Blue green to reddish brown

<b>Burette reading</b>	<b>I</b>	<b>II</b>	<b>III</b>
Final Reading			
Initial Reading			
Vol. of FAS run down in cm <sup>3</sup>			

Concordant burette reading-----

**Calculation:**

Vol. of FAS required to oxidize the oxidizable matter present in waste water (V<sub>oxidizable matter</sub>) = Blank titre value - Back titre value

$$(NV)_{\text{oxidizable matter}} = (NV)_{\text{FAS}}$$

$$(N)_{\text{Oxidizable matter}} = \frac{(NV)_{\text{FAS}}}{V_{\text{oxidizable matter}}} = \frac{\quad}{25} \text{ N}$$

Weight of oxidizable matter in 1 litre of water sample = N × Equivalent weight of oxygen (8)

$$W = N \times 8 \text{ g/litre}$$

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

$$\text{Amount of oxidizable matter in } 10^6 \text{ ml of the given water sample} = \frac{W \times 10^6}{1000} = \text{----- mg/litre}$$

**Result:** The COD of given waste water sample = ----- mg of O<sub>2</sub>/litre or ppm

## EXPERIMENT-5

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

#### Principle:

Chemical oxygen demand is a measure of the oxygen equivalent of organic and inorganic materials in a water sample that can be oxidized by a strong chemical oxidant. The parameter is important for industrial waste studies and to control waste treatment plants. COD is expressed as mg of oxygen required to oxidize impurities in  $1000\text{ cm}^3$  of waste water.

The oxidisable constituent of waste water includes straight chain aliphatic compounds, aromatic hydrocarbons, straight chain alcohols, acids, pyridine and other oxidisable materials. Straight chain compounds and acetic acid are oxidized more effectively in presence of silver ions (added as silver sulphate) as catalyst. However, the silver ions become ineffective in presence of halide. This difficulty is overcome by treating the waste water with mercuric sulphate before the analysis for COD. Mercuric sulphate binds the halide ions and makes them unavailable.

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

#### Procedure:

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

Weigh about 9.8 g of Mohr's salt accurately into a  $250\text{ cm}^3$  volumetric flask. Add half test tube of 1:1 sulphuric acid and dissolve the crystals. Dilute the solution with ion exchange water upto the mark and shake well.

##### Back Titration:

Pipette out  $25\text{ cm}^3$  of the waste water sample into a conical flask. Add  $10\text{ cm}^3$  of standard potassium dichromate solution followed by 1 test tube of 1:1 sulphuric acid (containing silver sulphate and mercuric chloride) with constant shaking. 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  $10\text{ cm}^3$  of standard potassium dichromate solution. Add 1 test tube of 1:1 sulphuric acid (containing silver sulphate and mercuric chloride) followed by 2-3 drops of ferroin indicator. Titrate against standard Mohr's salt solution until the color turns from bluish green to reddish brown.

**OBSERVATION & CALCULATIONS**

Burette readings	I	II	III
Initial readings			
Final readings			
Volume of $\text{Na}_2\text{S}_2\text{O}_3$ run down in ml			

**Calculation:**

Concordant burette reading-----

$$(NV)_{\text{H}_2\text{O}} = (NV)_{\text{Na}_2\text{S}_2\text{O}_3}$$

$$(N)_{\text{H}_2\text{O}} = \frac{(NV)_{\text{Na}_2\text{S}_2\text{O}_3}}{V_{\text{H}_2\text{O}}} = \frac{\text{-----}}{50} N$$

Weight of DO in one litre of water sample =  $N \times \text{Equivalent weight of oxygen (8)}$ 

$$W = N \times 8 \text{ g/litre}$$

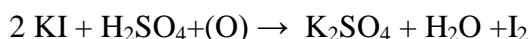
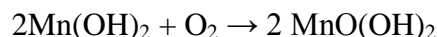
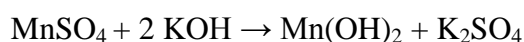
$$W = \text{-----g.}$$

Amount of DO in  $10^6$  ml of the given water sample =  $\frac{W \times 10^6}{1000} = \text{-----}$  mg/litre or ppm**Result:** Amount of dissolved oxygen in given sample of water = -----ppm

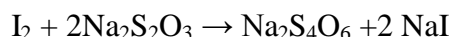


**EXPERIMENT-6****DETERMINATION OF DISSOLVED OXYGEN (DO) IN THE GIVEN WATER SAMPLE BY WINKLER'S METHOD****Principle:**

DO in water sample is determined by Winkler's method. This method is based on the fact that oxygen (dissolved in water) oxidizes KI to I<sub>2</sub>. The liberated iodine is titrated against a standard Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution as usual. However, since dissolved oxygen in water is in molecular state, it can't oxidize KI as such. Therefore, an oxygen carrier such as manganese hydroxide is used to bring about the reaction between KI and oxygen manganese hydroxide obtained by the action of KOH on manganese sulphate.



The iodine liberated is then titrated against standard sodium thiosulphate solution.

**Procedure:****Estimation of Dissolved Oxygen:**

Take 450 cm<sup>3</sup> of water sample in a BOD bottle. Add 3.0 cm<sup>3</sup> of manganous sulphate solution and 3.0 cm<sup>3</sup> of alkaline KI solution in a BOD bottle, stopper the bottle and shake thoroughly. When the precipitate is settled, add 3.0 cm<sup>3</sup> of conc. H<sub>2</sub>SO<sub>4</sub> and shake well until the precipitate has completely dissolved. Take 50ml of this solution and titrate against standard Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution using starch as indicator till blue colour disappears.

## **MODEL PROCEDURE FOR I/II SEMESTER CHEMISTRY PRACTICAL EXAMINATION**

## Part-A

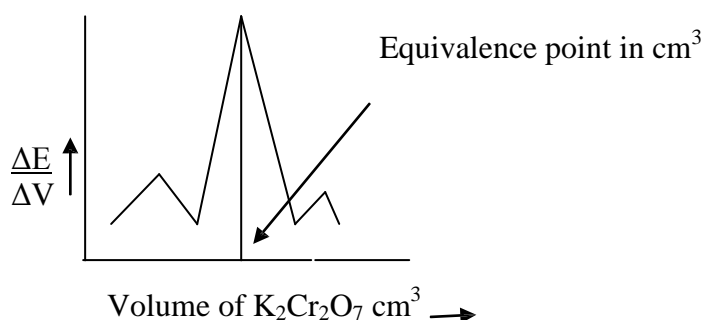
### INSTRUMENTAL EXPERIMENTS

#### Expt. 1: Estimation of FAS using standard $K_2Cr_2O_7$ solution by potentiometry

Take  $25\text{ cm}^3$  of FAS into a beaker, add 2 test tubes of  $H_2SO_4$  and immerse calomel electrode and platinum electrode assembly into it. Connect the assembly to a potentiometer and measure the potential. Add  $K_2Cr_2O_7$  from the burette in the increments of  $0.5\text{ cm}^3$  and measure the potential after each addition.

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

**Graph :**



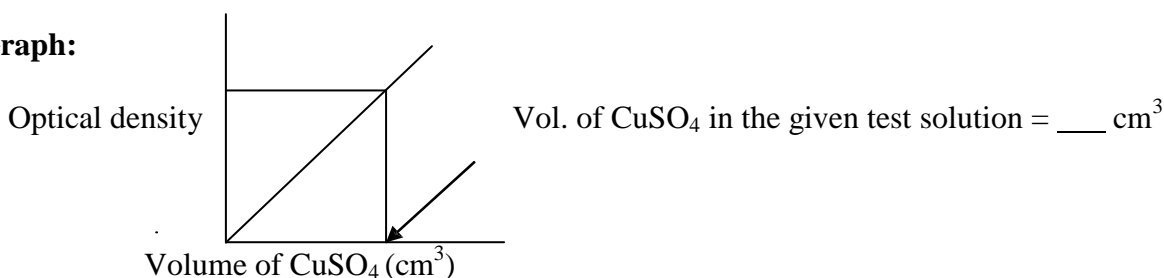
From the volume of  $K_2Cr_2O_7$  consumed, calculate weight of FAS in the given solution.

#### Expt.2: Estimation of copper in industrial waste water effluent by using colorimeter

Draw out  $5, 10, 15, 20$  and  $25\text{ cm}^3$  of given copper sulphate solution into 5 separate  $50\text{ cm}^3$  volumetric flask. Add  $5\text{ cm}^3$  of  $NH_3$  to each of them and also test solution, dilute upto the mark with distilled water and mix well. Measure the absorbance of each of the solution prepared against blank solution (only ammonia and water) at  $620\text{ nm}$ .

Plot a graph of optical density versus volume of copper sulphate.

**Graph:**



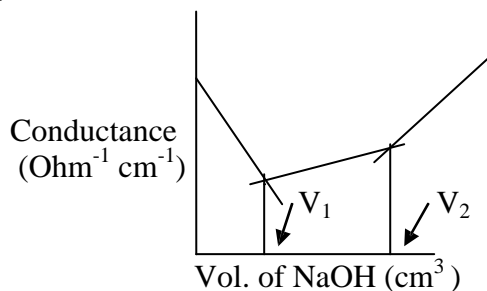
From the volume of copper sulphate, calculate the amount of copper present in the given test solution.

**Expt.3: Estimation of strength of an acid mixture by conductometry**

Take 50 cm<sup>3</sup> 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<sup>3</sup> and measure the conductance after each addition.

Plot a graph of conductance against (ordinate) volume of NaOH (abscissa) and find the equivalence points. From the volume of NaOH calculate the amount of HCl and CH<sub>3</sub>COOH in the given solution.

Graph:

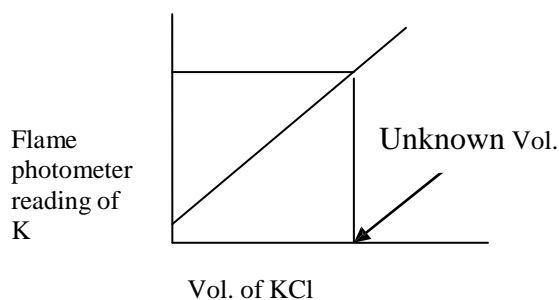
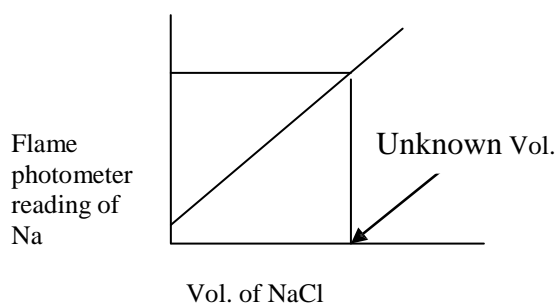


V<sub>1</sub> = Volume of NaOH consumed by HCl

V<sub>2</sub> = Volume of NaOH consumed by CH<sub>3</sub>COOH and HCl

**Expt. 4: Estimation of sodium and potassium by flame photometric method in a water sample**

Pipette out 5, 10, 15, 20 & 25 cm<sup>3</sup> of the given NaCl solution into the different 100 cm<sup>3</sup> volumetric flasks. Dilute the solution upto the mark with distilled water and shake well for uniform concentration. Take the first standard solution in a small beaker and aspirate into the flame of the flame photometer. The flame emission intensity of the standard is measured by the flame photometer using sodium filter (589 nm) in the photometer. The same is repeated for all the standard solutions with distilled water being sprayed to the flame in between the trials. Similarly, the flame emission intensity of standard and other solutions of KCl is measured by the flame photometer reading using potassium filter (766.5 nm). Plot the calibration curve by plotting emission response along y-axis and volume of the solution along the x-axis. From the calibration curve compute the concentration of Na and K in the water sample.

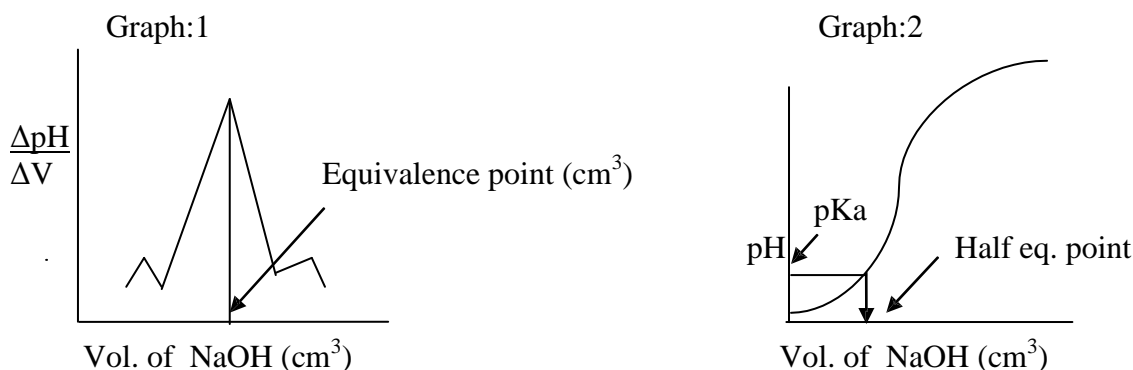


**Expt.5: Determination of  $pK_a$  value of a weak acid using pH meter**

Take 25 cm<sup>3</sup> of weak acid into a beaker. Immerse the glass and calomel electrodes into it. Connect the electrodes to pH meter and measure the pH. Now add NaOH from the burette in increments of 0.5 cm<sup>3</sup> and measure the pH after each addition.

Plot a graph of  $\Delta pH/\Delta V$  against volume of NaOH and find the equivalence point.

Plot another graph of pH against volume of NaOH and determine  $pK_a$  value of given weak acid at half equivalence point.

**Expt.6: Determination of viscosity co-efficient of a given lubricant/polymer using Ostwald's viscometer**

Pipette out 20 cm<sup>3</sup> of given liquid into a wide limb of the viscometer and suck the liquid through the other limb. Determine the time of flow between 2 fixed points, one above and one below the bulb in the narrow limb of the viscometer. Repeat and calculate the average time of flow.

Pour out the liquid, wash the viscometer with acetone and dry it. Now pipette out 20 cm<sup>3</sup> water into the wide limb and determine the average time of flow for water as before. By knowing the flow time of liquid and water, values of density of liquid and water and by using the viscosity coefficient of water, determine the viscosity coefficient of the given liquid.

Using Formula:  $\eta_l = \frac{t_l d_l}{t_w d_w} \times \eta_w$  milli poise

$t_l$  = time of flow of liquid

$d_l$  = density of liquid

$\eta_l$  = viscosity coefficient of liquid

$t_w$  = time of flow of water

$d_w$  = density of water

$\eta_w$  = viscosity coefficient of water

## Part-B

### VOLUMETRIC EXPERIMENTS

#### Expt.1: Assessment of total hardness in a sample of water by complexometric method

Known weight of disodium salt of EDTA is transferred into a 250 cm<sup>3</sup> volumetric flask and add ¼ test tube of ammonia. Dissolve with ion exchange water and dilute upto the mark, mix well.

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

**Burette** : Standard EDTA solution

**Conical flask** : 25cm<sup>3</sup> hard water sample + 2 cm<sup>3</sup> NH<sub>3</sub>-NH<sub>4</sub>Cl buffer solution (pH=10)

**Indicator** : Eriochrome Black –T

**Color change** : Wine red to clear blue

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

#### Expt. 2: Determination of Calcium oxide in the given sample of cement by rapid EDTA method

A known weight of disodium salt of EDTA is transferred into 250 cm<sup>3</sup> volumetric flask and add ¼ test tube of ammonia. Dissolve with ion exchange water and dilute upto the mark, mix well.

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

**Burette** : Standard EDTA solution

**Conical flask** : 25 cm<sup>3</sup> cement solution + 2 cm<sup>3</sup> of 1:1 glycerol + 2 cm<sup>3</sup> diethyl amine (pH = 12 to 14) + 2 cm<sup>3</sup> of 4 N NaOH

**Indicator** : Patton and Reeder's indicator

**Color change** : Wine red to clear blue

From the volume of EDTA consumed, calculate the % of CaO present in the given cement solution.

**Expt.3: Determination of percentage of copper in brass solution by Iodometric method.**

**Preparation of brass solution:** Dilute the given brass solution in a 250 ml Std. flask, add distilled water upto the mark and mix well.

**Burette :** Standard  $\text{Na}_2\text{S}_2\text{O}_3$  solution.

**Conical flask :** 25 ml of brass solution +  $\text{NH}_4\text{OH}$  till formation of blue colour + dil.  $\text{CH}_3\text{COOH}$  till disappearance of blue colour + 1 test tube of 10% KI solution.

**Indicator :** 2  $\text{cm}^3$  starch towards the end point (when the solution turns pale yellow)

**End point :** Disappearance of blue colour (formation of white ppt.)

From the volume of  $\text{Na}_2\text{S}_2\text{O}_3$  consumed, calculate the % of copper in the given brass solution

**Expt.4: Determination of iron in the given sample of rust solution by external indicator method**

Weigh out the given  $\text{K}_2\text{Cr}_2\text{O}_7$  crystals accurately and transfer it into a 250  $\text{cm}^3$  volumetric flask. Dissolve with ion exchange water and dilute upto the mark and mix well.

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

**Burette:** Standard  $\text{K}_2\text{Cr}_2\text{O}_7$  solution

**Conical flask:** 25  $\text{cm}^3$  Haematite ore solution +  $\frac{1}{4}$  test tube conc.  $\text{HCl}$  and boil +  $\text{SnCl}_2$  drop wise till colourless + cool +  $\frac{1}{4}$  test tube  $\text{HgCl}_2$  (Silky white Precipitate is formed) + 1 test tube of water.

**Indicator:**  $\text{K}_3\text{Fe}(\text{CN})_6$  - External indicator (Potassium Ferricyanide)

**End Pont:** Failure to develop blue colour with the test drop

From the volume of  $\text{K}_2\text{Cr}_2\text{O}_7$  consumed, calculate the % of Iron in the given haematite ore solution

**Expt. 5: Determination of chemical oxygen demand (COD) of the given industrial waste water sample**

Weigh out given FAS crystals accurately and transfer into a 250 cm<sup>3</sup> of volumetric flask, add 1/2 test tube of dilute H<sub>2</sub>SO<sub>4</sub>, dissolve the crystals, dilute upto the mark with ion exchange water and mix well.

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

**Burette** : Standard FAS solution

**Conical flask:** 25 cm<sup>3</sup> waste water + 10 cm<sup>3</sup> K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> + 1 test tube of 1:1 H<sub>2</sub>SO<sub>4</sub>, boil for half an hour, cool

**Indicator** : Ferroin

**Colour change** : Bluish green to reddish brown

**Blank Titration** : It is performed same as above without waste water sample.

From the difference in the titre values, calculate the COD of the given waste water sample.

**Expt.6: Determination of dissolved oxygen (DO) in the given water sample by Winkler's method****Preparation of water containing free iodine**

Take 450 ml of water sample in a BOD bottle + 3ml of MnSO<sub>4</sub> + 3ml of alkaline KI solution + 3ml of con. H<sub>2</sub>SO<sub>4</sub>, stopper the bottle and mix well.

**Burette:** Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution

**Conical flask:** 50ml of water containing free Iodine

**Indicator:** Starch near the end point

**End Point:** Disappearance of blue color

From the volume of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution consumed, calculate the amount of dissolved oxygen in the given water sample.



**MODEL  
VIVA –VOCE QUESTIONS  
WITH ANSWERS**

**Part-A****INSTRUMENTAL EXPERIMENTS****Expt.-1: Estimation of FAS using standard  $K_2Cr_2O_7$  solution by potentiometry**

1. What is potentiometric titration?

The determination of equivalence point of redox titrations on the basis of potential measurements is called a potentiometric titration.

2. Give the principle of potentiometric titration?

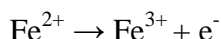
The principle involved in potentiometric titration is the measurement of the emf between two electrodes, an indicator electrode and a reference electrode. In these titrations, measurements of emf are made while the titration is in progress. The equivalence point of the reaction is revealed by a sudden change in potential in the plot of emf readings against the volume of titrant.

3. What are the electrodes used in potentiometric titration?

The indicator electrode is the platinum electrode and the reference electrode used is the calomel electrode.

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

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



5. What is an indicator electrode?

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

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

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



7. What are the advantages of potentiometric titration?

- i. Turbid, fluorescent, opaque or colored solutions can be titrated.
- ii. Mixture of solutions or very dilute solutions can be titrated.
- iii. The results are more accurate because the actual end point is determined graphically.

## Expt.-2: Estimation of copper in industrial waste water effluent by using colorimeter

1. What is colorimetry?  
Chemical analysis through measurements of absorption of light radiation in the visible region of the spectrum (400-760 nm) with respect to a known concentration of the substance is known as colorimetry.
2. What forms the basis of colorimetric Determination?  
The variation of color of a system with change in concentration of complex forms the basis of the colorimetric determination.
3. What is photoelectric colorimeter?  
It is electrical instrument, which measures the amount of light absorbed using a photocell.
4. What are filters? Why are they used?  
The filters consist of either thin films of gelatin containing different dyes or glass. The filters are used in colorimeters for isolating (selecting) any desired spectral region.
5. State Beer's law?  
The intensity of a beam of monochromatic light decreases exponentially as the concentration of the absorbing substance increases arithmetically.
6. 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.
7. State Beer-Lambert's law?  
The amount of light absorbed is directly proportional to the concentration of the solution.  $A = \log I_0 / I_t = \epsilon C t$  Where ' $\epsilon$ ' is the molar extinction coefficient, 'C' is the concentration and 't' is the path length.
8. What is a calibration curve?  
It is the plot of optical density (absorbance) against known concentration of solutions. For solutions obeying Beer's law, this is a straight line.
9. What is meant by transmittance?  
It is the ratio of amount of light transmitted to the total amount of light absorbed
10. Mention advantages of photoelectric colorimetric determinations?
  - i A colorimetric method will often give more accurate results at low concentration than the corresponding titrimetric or gravimetric procedure.
  - ii A colorimetric method may frequently be applied where no satisfactory gravimetric or titrimetric procedure exists i.e., for certain biological substances.
11. What is a blank solution?  
A blank solution is identical in all respects to the test solution except for the absence of test solute.
12. Why is ammonia added?  
Ammonia is added to get cuprammonium sulphate complex,  $[\text{Cu}(\text{NH}_3)_4] \text{SO}_4$  a dark blue complex.
13. Why estimations of copper are read at 620 nm wavelength?  
The estimation of copper are carried out at 620 nm wavelength because the complex shows a maximum absorbance at 620 nm.

**Expt.-3: Estimation of strength of an acid mixture by conductometry**

1. State Ohm's law.  
Ohm's law states that the current  $I$  (ampere) flowing in a conductor is directly proportional to the applied electromotive force and inversely proportional to the resistance  $R$  (ohm) of the conductor.
2. What is conductance?  
The reciprocal of resistance is called conductance.
3. What is the unit of conductance?  
The unit of conductivity is  $\text{Ohm}^{-1} \text{cm}^{-1}$  or  $\text{Sm}^{-1}$ .
4. Mention the different types of conductivity.  
A solution may have following conductivity: i) Specific conductivity ii) Equivalent conductivity iii) Molar conductivity
5. Which of the above conductivities measured during conductometric titration?  
The specific conductivity is measured.
6. What is specific conductivity?  
It is the conductivity of a solution between electrodes of  $1 \text{ cm}^2$  area and kept  $1 \text{ cm}$  apart.
7. What is equivalent conductivity?  
It is the conductivity of a solution which contains  $1$  gram equivalent of solute, when placed between two electrodes of  $1 \text{ cm}$  apart.  

$$\lambda = kV$$

$$\lambda = \frac{k \times 1000}{C}$$

$$V = \text{volume containing } 1 \text{ gram equivalent of solute}$$

$$C = \text{Normality of solution.}$$
8. What is molar conductivity?  
It is the conductivity of a solution which contains  $1$  gram molecular weight of a substance, when placed between two electrodes of  $1 \text{ cm}$  apart.  

$$\mu = kV$$
9. What is the principle involved in conductometric titration?  
In conductometric titrations, there is a sudden increase in conductance of the solution near the end point. Hence the end point is determined graphically by Plotting conductance against titer values. The principle underlying conductometric titrations is the replacement of ions of a particular conductance by ions of another conductance during titration.  

$$\text{NaOH} + \text{HCl} \rightarrow \text{NaCl} + \text{H}_2\text{O}$$
10. Explain the variation in conductivity at the end point ?  
The addition of sodium hydroxide to hydrochloric acid decreases the conductance because of replacement of highly mobile  $\text{H}^+$  ions (conductivity  $350 \text{ Ohm}^{-1} \text{cm}^{-1}$ ) by the less mobile  $\text{Na}^+$  ions (conductivity  $50 \text{ Ohm}^{-1} \text{cm}^{-1}$ ). This trend continues till all the hydrogen ions are replaced and the end point is reached. A further addition of sodium hydroxide increases the conductance because of the presence of free  $\text{OH}^-$  ions (Conductivity:  $198 \text{ Ohm}^{-1} \text{cm}^{-1}$ ).
11. 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  $\text{H}^+$  ions. As a result, the magnitude of decrease in conductivity is more before the neutralization point Compared to the magnitude of increase after the neutralization

point. Hence the slopes of the curves before and after the neutralization point are not exactly the same.

12. How is accuracy of the method determined?

The accuracy of the method is greater when the angle of intersection is more accurate and more nearly the points of the graph on a straight line.

13. What are the advantages of conductometric titrations?

i The method is accurate in dilute as well as more concentrated solutions.

ii It can also be employed with coloured solutions.

iii Very weak acids such as  $\text{H}_3\text{PO}_3$ , phenol, which cannot be titrated potentiometrically in aqueous solutions can be titrated conductometric.

iv Mixture of acids can be titrated more accurately.

#### Expt.-4: Estimation of sodium and potassium by flame photometric method in a water sample

1. Why sodium and potassium are estimated by flame photometry?

Alkali metals generally have lower ionization energies and can easily be excited to higher energy states. Emission spectrums of such elements are of use to estimate them quantitatively

2. What is flame photometry?

In flame photometry, atomic emission technique is used for the estimation of the Na, K and other metals in a given metal salt solution

3. What is basic principle involved in Flame Photometric technique?

In Flame Photometry, the intensity of emission radiation is correlated to the concentration of the metal that is being estimated in the given sample of metal salt.

4. What is an optical filter in Flame Photometry?

Optical filters are used to select the emission wave length monitor for the analyte species.

5. How does a flame photometer work?

In the flame photometric technique, solutions are aspirated into the flame. The hot flame evaporates the solvent, atomizes the metal, and excites a valence electron to an upper state. Light is emitted at characteristic wavelength for each metal as electron returns to ground state, it is measured using a photo detector.

#### Expt.-5: Determination of $\text{pK}_a$ value of a weak acid using pH meter

1. What is a weak acid?

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

2. What is  $\text{pK}_a$  of weak acid?

$\text{pK}_a$  is a modern method of expressing the strength of weak acids is mathematically expressed as  $\text{pK}_a = -\log_{10} K_a$ . where  $K_a$  is the dissociation constant of the weak acid.

3. What is meant by pH solution?

$\text{p}^{\text{H}}$  of a solution as negative logarithm to base 10 of hydrogen ion concentration.

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

4. Why is a glass electrode called as ion selective electrode?

The glass electrode called an ion selective electrode because, it is able to respond to certain specific ions ( $\text{H}^+$  ions) only and develop a potential while ignoring the other ions in the solution.

5. How is the measurement of pH made?

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

6. How are pH and  $\text{pK}_a$  related?

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

At half equivalence point  $[\text{salt}] = [\text{acid}]$ . Therefore,  $\text{p}^H$  at half equivalence gives the  $\text{pK}_a$  of the weak acid.

7. How are  $\text{pK}_a$  and strength of a weak acid related?

Higher the  $\text{pK}_a$ , lower will be the strength of the weak acid.

8. What are the electrodes used in the measurement of pH for the determination of  $\text{pK}_a$ ?

In the determination of  $\text{pK}_a$  of a weak acid glass electrode (ion selective electrode) and calomel electrode (reference electrode) are used. In this case, glass electrode acts as an anode and calomel electrode acts as a cathode.

9. 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 resulting in sudden increase of  $\text{p}^H$ .

#### Expt.-6: Determination of viscosity co-efficient of a given lubricant/polymer using Ostwald's viscometer

1. What is viscosity?

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

2. What is viscosity coefficient of a liquid?

The viscosity coefficient of a liquid is defined as the tangential force per unit area required to maintain a unit velocity gradient between any two successive layers of a liquid situated unit distance apart.

What is density of a liquid?

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

What is the SI unit of viscosity coefficient?

The viscosity coefficient is expressed as Pascal second (Ps)  $1 \text{ Ps} = 1 \text{ kg m}^{-1} \text{ s}^{-1}$ .

4. What is the equation used to represent the influence of temperature on viscosity?

The influence of temperature on viscosity is best represented by an empirical equation  $\eta = Ae^{B/RT}$  where A and B are constants for a given liquid.

5. What are the factors that affect the viscosity of a liquid?

- Increase in molecular Weight results in an increase in viscosity.
- Branched chain compounds have higher viscosity than those involving straight chain compounds.
- The polar compounds are more viscous than non polar ones. The presence of hydrogen bonds cause the viscosity to increase.
- The viscosity of liquids increases by the presence of lyophilic colloids and suspended impurities.
- Temperature has marked influence on the viscosity of a liquid (2% per degree).

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

The law based on the viscosity flow of liquids through capillary tubes is Poissioeuille law. It is expressed as

$$\eta = \frac{\pi r^4 \Delta p}{8 \nu l}$$

$\eta$  = viscosity coefficient;  $r$  = radius of the tube.  $t$  is the time required for the volume,  $V$  is the volume of liquid to flow through the tube of length 'l'

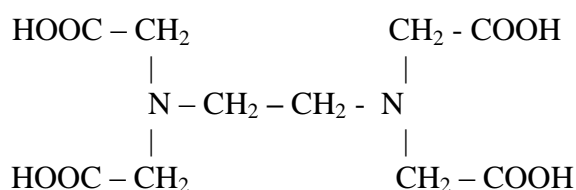
7. How does the viscosity vary with temperature?  
The viscosity of a liquid usually decreases with the rise of temperature.
8. Why should the viscometer be dried before the measurements are done?  
The viscometer should be dried to avoid the formation of emulsion, which changes the rate of flow of the liquid.
9. Why is acetone used for cleaning viscometer?  
Acetone is a volatile organic liquid. To dry the viscometer quickly, it is rinsed with acetone.
10. Why is viscometer not rinsed with the given liquid or water?  
If the viscometer is rinsed with the given liquid or water before measuring the flow time, the volume taken will be more than a definite known volume.
11. How is the viscosity of a liquid related to its mobility?  
Viscosity of a liquid is inversely proportional to its mobility.

## Part-B VOLUMETRIC

### EXPERIMENTS

#### Expt.-1: Assessment of total hardness in a sample of water by complexometric method

1. What is hard water?  
The water which does not produce lather easily and readily with the action of soap is called hard water.
2. What are the types of hardness of water?  
Temporary hardness water  
Permanent hardness water
3. How is hardness of water caused?  
Hardness of water is caused by divalent metallic cations in association with anions such as  $\text{HCO}_3^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{Cl}^-$ ,  $\text{SiO}_3^{2-}$ , etc
4. How is temporary hardness removed?  
Temporary hardness of water can be removed by boiling the water during which bicarbonates decompose to give carbonates.
5. How do you express the total hardness of water?  
It is expressed as parts per million of  $\text{CaCO}_3$ .
6. What is EDTA?  
Ethylene diamine tetra acetic acid.
7. Write the structural formula of EDTA?



8. Why is disodium salt of EDTA preferred to EDTA?  
EDTA is sparingly soluble in water. Therefore, its disodium salt which is more soluble is preferred.

9. Why is ammonia solution added while preparing EDTA solution?  
Ammonia solution is added while preparing EDTA solution to increase the rate of dissolution of the salt.
10. What is buffer solution?  
The solution which resists change in its  $p^H$  value even after adding small amount of an acid or base to it, is called a buffer solution.
11. Why is ammonia- ammonium chloride buffer added?  
To maintain a  $pH$  of 10, the desired  $pH$  for the titration.
12. Why does the indicator Erichrome Black – T (EBT) show wine red colour at the beginning and blue at the end?  
When a small amount of Erichrome Black –T which is blue in colour, is added to water sample at  $P^H$  of about 10.0, it combines with a few of the calcium and magnesium ions to form a weak complex which is wine red colour as shown in eq.  
$$M^{2+} + EBT \longrightarrow (M- EBT)$$
  
Wine red complex  
During the titration with EDTA, all free hardness causing ions are complexed according to eq.  
$$M^{2+} + EDTA \longrightarrow (M-EDTA)$$
  
Stable complex  
This action frees the EBT indicator from the complex. (M- EBT), and the wine red colour changes to a clear blue colour at the end of the titration.
13. Why are the titrations involving EDTA carried out slowly towards the end point?  
The rate of formation of the metal complex of EDTA is very slow.
14. What is the application of hardness data in environmental engineering practice?
  - Hardness of water is an important consideration in determining the suitability of a water for domestic and industrial uses.
  - Determination of hardness serves as a basis for routine control of softening processes.
15. What is the chemical name of Erichrome black – T?  
Sodium 1 – (1-hydroxy-2-naphthylazo)-6-nitro-2-naphthol-4-sulphonate (SHNNNS)

### Expt.-2: Determination of Calcium oxide in the given sample of cement by rapid EDTA method

1. What are the constituents of cement?  
Oxides of calcium, magnesium, iron, aluminum and silicon.
2. What is the prime constituent of cement?  
Calcium oxide
3. What is the role of glycerol?  
Glycerol is added to get the sharp end point.
4. What is the role of NaOH?  
NaOH is added to mask the magnesium ion which interrupts during the course of titration.
5. What is the function of diethyl amine?  
Diethyl amine is added to maintain  $pH$  of about 12.5
6. Why EBT indicator is cannot be used in this Experiment?  
EBT forms a very weak complex with calcium ions at a  $pH$  range of 12-14.
7. Which indicator is used in determination of CaO in cement solution?  
'Patton and Reeder's indicator'



8. What is the chemical name of 'Patton and Reeder' solution indicator?  
2-Hydroxy-1-(2-hydroxy-4-sulfo-1-naphthylazo)-3-naphthoic acid.
9. Why this titration is called rapid EDTA method?  
The titration is called rapid EDTA method because, by this method  $\text{Ca}^{2+}$  ions in cement solutions are estimated directly and quickly by using standard EDTA solution without removing the other metal ions.

### Expt.-3: Determination of percentage of copper in brass solution by Iodometric method

1. What are the constituents of brass?  
Brass is an alloy of copper (50-90%) and Zinc (20-40%). It also contains small quantities of tin and iron. The percentage composition of a typical brass alloy is Cu; 50-90%, Zn; 20-40%, Sn; 0-6%, Pb; 0-2%, Fe; 0-1%.
2. How is brass solution prepared?  
Brass solution prepared by dissolving the brass foils in 1:1 nitric acid.
3. What is purpose of adding urea?  
Urea is added to remove all oxides of nitrogen
4. Why is ammonium hydroxide added to brass solution?  
To neutralize the mineral acid ( $\text{HNO}_3$ ).
5. What is the bluish white precipitate formed after adding ammonia solution?  
A bluish white precipitate of cupric hydroxide  $\text{Cu}(\text{OH})_2$  is formed, when ammonia solution is added to the brass solution
6. Why is acetic acid added?  
Acetic acid is added to neutralize the excess of ammonia hydroxide and to make solution slightly acidic.
7. Why is potassium iodide is added to brass solution although Cu in brass is determined?  
Cupric ions do not react with sodium thiosulphate solution. However, cupric ions Oxidize potassium iodide and iodine is liberated. The amount of iodine liberated is equal to the amount of cupric ions present in solution.  

$$2\text{Cu}^{2+} + 4\text{KI} \longrightarrow \text{Cu}_2\text{I}_2 + 4\text{K}^+ + \text{I}_2$$
8. How is liberated iodine estimated?  
The amount of iodine liberated is estimated by titrating it against standard sodium thiosulphate solution using starch as an indicator.
9. What is the reaction that occurs between iodine and sodium thiosulphate?  
Iodine reacts with sodium thiosulphate to form sodium iodide and sodium tetrathionate which are colorless products.  

$$2\text{Na}_2\text{S}_2\text{O}_3 + \text{I}_2 \longrightarrow \text{Na}_2\text{S}_4\text{O}_6 + 2\text{NaI}$$
10. Why is starch indicator added towards the end point?  
If the starch indicator added at the beginning the intensity of the iodine is more, the starch indicator forms a stable water insoluble complex with iodine as a result the volume of sodium thiosulphate consumed will be less than expected.
11. What is white precipitate produced at the end of the point?  
The white precipitate produced at the end of the point is cuprous iodide ( $\text{Cu}_2\text{I}_2$ )

**Expt.-4: Determination of iron in the given sample of rust solution by external indicator method**

1. What are main constituents of haematite ore?

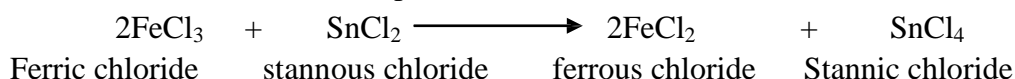
Haematite is an important ore of iron containing mainly  $\text{Fe}_2\text{O}_3$  and small amount of silica ( $\text{SiO}_2$ ).

2. Give the other forms of iron ore?

Magnetite ( $\text{Fe}_3\text{O}_4$ ), Siderite or Spathic iron ( $\text{Fe}_2\text{O}_3$ ), iron pyrites ( $\text{FeS}_2$ ), etc.

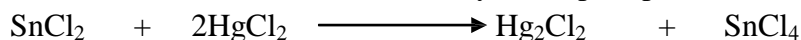
3. What is the role of stannous chloride?

Stannous chloride reduces ferric ions ( $\text{Fe}^{+++}$ ) in to ferrous ( $\text{Fe}^{++}$ ) ions and a slight excess is added to ensure complete reduction.



4. Why is mercuric chloride added?

Mercuric chloride is added to remove excess stannous chloride. Mercuric chloride reacts with stannous chloride to form silky white precipitate of mercurous chloride.



5. What happens when the excess of stannous chloride is not removed?

If the excess of stannous chloride is not removed, the ferric ions formed during the course of titration get reduced to ferrous ion. As a result the volume of titrant, Potassium dichromate consumed will be more than expected.

6. What is indicator used?

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

7. Why potassium ferricyanide can't be used as internal indicator in analysis of haematite?

Potassium ferricyanide combines irreversibly with ferrous ions to form a deep blue ferrous ferricyanide complex (Tumbull's blue). Thus ferrous ions involved in complex formation are not available for reaction with potassium dichromate. Moreover, the end point can't be detected as there is no colour change.

8. Why potassium ferrocyanide can't be used as indicator in estimation of iron?

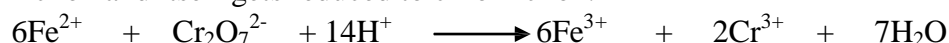
Potassium ferrocyanide can't be used as it does not react with ferrous ion.

9. Why the colour of indicator drop remains same at the end point?

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

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

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



**Expt.-5: Determination of chemical oxygen demand (COD) of the given industrial waste water sample**

1. What is chemical oxygen demand?  
Chemical oxygen demand is the 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.
2. What general groups of organic compounds are not oxidized in the COD test?  
Aromatic hydrocarbons and pyridine are not oxidized in COD test.
3. What is the role of silver sulphate?  
Silver sulphate acts as a catalyst in oxidation of straight chain aliphatic hydrocarbons, and oxidation is effective in the presence of silver ions.
4. What is the role of mercuric sulphate?  
Chloride ions normally present in high concentration in waste water which undergo oxidation in COD test and causes 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.
5. 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.
6. Why is sulphuric acid added during the preparation of standard FAS solution?  
Sulphuric acid added to prevent the hydrolysis of ferrous sulphate into ferrous hydroxide.
7. What is composition of ferroin?  
Ferrous 1, 10-phenanthroline sulphate.
8. Mention a few application of COD test in environmental engineering practice.
  - i. COD test is extensively used in the analysis of industrial wastes.
  - ii. COD test is helpful in indicating toxic conditions and the presence of biologically resistant organic substances.
9. What is the limitation of COD?  
One of the chief limitation of COD test is its 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 exist in nature.

**Expt.-6: Determination of dissolved oxygen (DO) in the given water sample by Winkler's method**

1. What is meant by dissolved oxygen?

Water found in nature contains a small amount of oxygen in it called dissolved oxygen.

2. What is the importance of dissolved oxygen in the given sample of water?

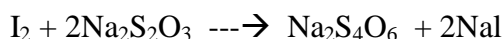
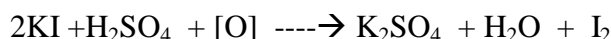
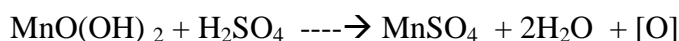
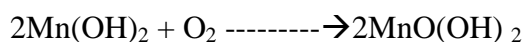
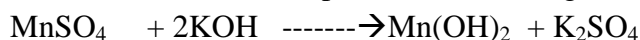
The analysis of DO is a key test to assess the raw water quality and to keep a check on stream pollution. DO forms the basis of BOD which constitutes an important parameter to evaluate pollution, Potential of wastes. Dissolved oxygen test is used to control the amount of oxygen in boiler feed water by physical, chemical and mechanical methods.

3. Differentiate between dissolved oxygen and COD.

DO is the measure of oxygen dissolved in water, it gives the available oxygen in the water. COD is a measure of oxidisable impurities (both biologically oxidisable and biologically inert organic matter) present in the water sample. It gives the oxygen demand of the water sample.

4. Explain the principle involved in Winkler's method.

The principle involved in the determination of DO is bring about the oxidization of KI to iodine with dissolved oxygen present in the water sample after adding  $\text{MnSO}_4$ , KI, the basic manganese oxide formed acts as oxygen carrier to enable the dissolved oxygen in the molecular form to take part in the reaction. The liberated Iodine is titrated against standard sodium thiosulphate solution using starch as indicator.

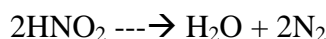
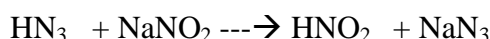
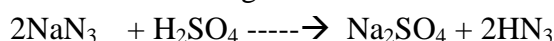


5. What are iodometric titrations?

Titration involving liberation of iodine are called iodometric titrations.

6. What is the function of sodium azide in the estimation of water?

If nitrites, sulphates are present in water, they also oxidize KI to  $\text{I}_2$  during estimation of dissolved oxygen and leads to wrong results. Therefore azide modification is used for most of the effluent and sewage.



7. Upon what factors does dissolution of oxygen in water depend?

The amount of oxygen that dissolved in water depends on the following factors.

- i. Adequate time and adequate mixing to fully saturate the water.
- ii. The water temperature.
- iii. The air pressure
- iv. The salt content of the water.
- v. The substances in water which consume oxygen.

8. How does the presence of  $\text{NO}_2$  affect the dissolved oxygen result?

In the modified Winkler's method, the interference due to certain oxidizing agent such as  $\text{NO}_2$  or reducing agent such as  $\text{Fe}^{2+}$  or  $\text{SO}_3^{2-}$  is removed by treating the sample with excess of  $\text{KMnO}_4$  in acid medium.

9. What is the effect of oxidisable impurities on the dissolution of oxygen?

Oxidisable impurities get oxidized on the dissolution of oxygen.

10. When do you say the sample of water is polluted badly?

Greater the BOD value, larger is the concentration of the decomposable organic matter and consequently greater will be the potential of the water / sewage.

11. The reaction is carried out in a BOD bottle and not in a conical flask. Why?

In order to prevent the oxidization of the reaction mixture by air, the reaction is carried out in a BOD bottle.

12. Define BOD and COD of water.

BOD is defined as the quantity of oxygen required by microorganisms to oxidize the organic wastes present in one liter of waste water over a period of five-days at  $20^\circ\text{C}$ . The unit of BOD is mg liter or ppm.

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