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# **GURU NANAK DEV ENGINEERING COLLEGE, BIDAR**

## **DEPARTMENT OF APPLIED SCIENCES & HUMANITIES**

### **Vision of the Institute**

To be a premier technological institution that fosters humanity, ethics and excellence in education and research towards inspiring and developing future torch bearers/leaders.

### **Mission of the Institute**

- M1** To impart quality educational experience and technical skills to students that enables them to become leaders in their chosen professions.
- M2** To nurture scientific temperament and promote research and development activities among faculty and students.
- M3** To inculcate students with an ethical and human approach, so as to have big picture of societal development in their future career.
- M4** To provide service to industries and communities through educational, technical, and professional activities.

### **Vision of the Department**

To be a vibrant department providing both foundational and finishing touches towards successful professional engineers known for its innovative teaching learning process and research.

### **Mission of the Department**

- M1** To impart strong foundational knowledge in applied science required for aspiring under-graduate engineering students.
- M2** To impart sound communication and managerial skills required for a successful engineering professional.
- M3** To promote research culture and innovative teaching learning process towards creation of new knowledge and improved learning.



## **Safety precautions / Do's and Dont's'**

### **DO's**

- Maintain discipline when you are in the laboratory.
- Adhere and follow timings, proper dress code with appropriate footwear.
- Bags, and other personal items must be stored in designated place.
- Come prepared with viva, procedure, and other details of the experiment.
- Always wear labcoat/Apron in the laboratory.
- Keep your face away from reaction vessel.
- Chemical reaction must be supervised.
- Learn to operate fire-extinguisher.
- Keep first –aid box within reach.
- Keep water and gas taps closed except when these utilities are needed.
- Inspect all equipment/apparatus for damage prior to use.
- Handle the apparatus/instruments 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.

### **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.
  - Do not add water to acid, add acid to water.
  - Do not take away glasswares or chemicals without instructor's permission.
  - Beware cold and hot glasses look alike.
- 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 observation book



## **LIST OF EXPERIMENTS**

### **1. Compulsory Experiments for all Branches in Exercise and Structured Enquiry B.E I / II Semester 2022-23 For Engineering Chemistry Lab**

- 1 . Potentiometric estimation of FAS using standard  $K_2Cr_2O_7$  solution.
2. Conductometric estimation of acid mixture.
3. Determination of Viscosity co-efficient of the given liquid using Ostwald's viscometer.
4. Colorimetric estimation of Copper.
5. Determination of pKa of the given weak acid using pH meter.
6. Estimation of Total hardness of water by EDTA complexometric method.
7. Determination of COD of waste water.
8. Estimation of iron in TMT bar by diphenyle amine.

### **2. Demo Experiments (For Electrical and Electronics Engineering Stream BCHEE102)**

9. Synthesis of Iron oxide Nanoparticles by precipitation method.
10. Determination of strength of Acid in lead-Acid battery.

### **3. Open Ended Experiments For Electrical and Electronics Engineering Stream BCHEE102)**

11. Estimation of Metal in e-waste by optical sensors.
12. Determination of glucose by electrochemical sensors.



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### EXPERIMENT NO.01

#### 1. POTENTIOMETRIC ESTIMATION OF FAS USING STANDARD $K_2Cr_2O_7$ SOLUTION

##### PRINCIPLE:

Redox titration can be carried out potentiometrically using platinum-calomel electrode combination in a manner similar to acid-base neutralizations for the reaction.

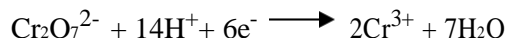
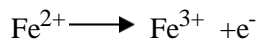
Reduced state  $\rightarrow$  oxidized state + n electrons

The potential is given by Nernst equation.

$$E = E^0 + \frac{0.0591}{n} \log \frac{(\text{oxidized state})}{(\text{reduced state})}$$

Where  $E^0$  is the standard potential of the system. The potential of the system is thus controlled by the ratio of the concentration of the oxidized to that of the reduced species present. As the reaction proceeds, the ratio increases and hence the potential changes more rapidly in the vicinity of the end point of the titration. This may be followed potentiometrically and a plot of change in potential against volume of potassium dichromate (titration curve) is characterized by a sudden change of potential at the equivalence point is explained as follows.

The reaction that takes place in the determination of  $Fe^{3+}$  is



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

$$E_{\text{cell}} = E^0 + \frac{0.0591}{n} \log \frac{(Fe^{3+})}{(Fe^{2+})}$$

Beyond the equivalence point the potential is determined by \_\_\_\_\_

$$E_{\text{cell}} = E^0_{Cr_2O_7^{2-} / Cr^{3+}} + \frac{0.0591}{6} \log \frac{(Cr_2O_7^{2-})}{(Cr^{3+})}$$

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

##### PROCEDURE:

Transfer 25ml of Ferrous sulphate solution into a beaker add 1 test tube full distilled water with help of pipette. Add 1 test tube of Dilute sulphuric acid. Immerse the electrode assembly into the solution in the beaker and connect the electrodes to a potentiometer. Measure the potential. Fill the burette with standard potassium dichromate solution.

Add 0.5 ml of potassium dichromate to the beaker. Stir the solution carefully and measure the potential after 15 seconds. Continue the procedure till potential shows a tendency to increase rapidly.

Determine the end point by differential method i.e. by plotting  $E / V$  against volume as shown in the figure. Calculate the normality of ferrous solution and determine the amount of iron in the given solution.



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### Tabular Column

Volume of Mohr's salt sample solution	V <sub>K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub></sub>	EMF E(MV)	ΔE	ΔV	ΔE/ΔV
25 ml FAS Sol'n + 1 test tube Full distilled water + 1 test tube full Dil sulphuric Acid.	0.0		--	--	--
	0.5			0.5	
	1.0			0.5	
	1.5			0.5	
	2.0			0.5	
	2.5			0.5	
	3.0			0.5	
	3.5			0.5	
	4.0			0.5	
	4.5			0.5	
	5.0			0.5	
	5.5			0.5	
	6.0			0.5	
	6.5			0.5	

### CALCULATION :

$$N_{FAS} = \frac{N_{K_2Cr_2O_7} \times V_{K_2Cr_2O_7}}{V_{FAS}}$$

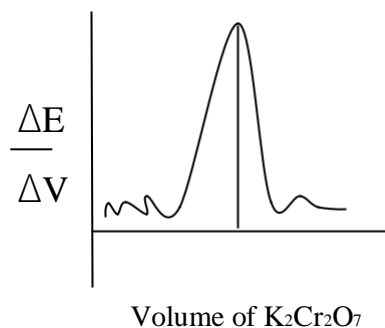
= 'X'

Amount of FAS in the given solution.

= 'X' x 392 gm/liter

=..... gm/liter

### Model Graph





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### **EXPERIMENT NO.02**

#### **2. ESTIMATION OF ACIDS IN ACID MIXTURE CONDUCTOMETRICALLY.**

##### **PRINCIPLE:-**

Conductance of a solution depends upon the number of ions and mobility of ions present in the solution. It is reciprocal of resistance. It represents the ease with which current flows through the conductor or electrolytic solution. Its unit is  $\text{ohm}^{-1}$  or mho or Siemens.

In conductometric titration, there is sudden increase in the conductance of the solution at equivalent point. The equivalent point is determined graphically by plotting conductance against the volume of base consumed.

In the titration of Acid mixture (Ex. Mix. Of  $\text{CH}_3\text{COOH}$  &  $\text{HCl}$ ) with strong base, the conductance decreases upon adding  $\text{NaOH}$  to Acid mixture due to substitution of highly mobile  $\text{H}^+$  ions by less mobile  $\text{Na}^+$  ions. This continues till the  $\text{H}^+$  ions of  $\text{HCl}$  get replaced i.e. strong acid is neutralized (first equivalence point). With continuous addition of  $\text{NaOH}$ , the conductance increases moderately as weak acid ( $\text{CH}_3\text{COOH}$ ) is now get converted into its salt ( $\text{CH}_3\text{COONa}$ ). On addition of  $\text{NaOH}$  further the conductance rises steeply due to presence of  $\text{OH}^-$  ions form  $\text{NaOH}$  as all the acid gets neutralized up to second equivalent point. The titration curves in the graph determine the location of equivalence points.

**PROCEDURE :-** Pipette out 50ml of Acid mixture in 100ml beaker. Dip the conductivity Cell and note down the Conductance of the solution without adding  $\text{NaOH}$  solution. Now add standard  $\text{NaOH}$  solution (0.1N) from the burette in increments of 0.5ml. After each addition, stir the solution gently and note down the Conductance As the titrations proceeds, the Conductance first gradually decreases and then rise slowly and finally rise sharply. Continue the titration till the conductance is more or less the same as it was in the beginning.

A graph is plotted between conductance (Y-axis) versus Volume of  $\text{NaOH}$  (x-axis) the point of intersection of first and second straight lines gives the volume of  $\text{NaOH}$  needed to neutralize only  $\text{HCl}$ . The point of intersection of second and third straight lines gives the volume of  $\text{NaOH}$  required to neutralize both  $\text{HCl}$  and  $\text{CH}_3\text{COOH}$ .



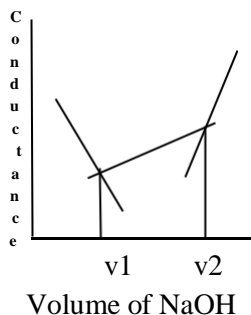
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### Observation and Calculation :

Sl.No.	Vol. of NaOH (0.1N)	Conductance ( $10^{-6} \text{ ohm}^{-1} \text{ cm}^{-1}$ )
1	0.0	
2	0.5	
3	1.0	
4	1.5	
5	2.0	
6	2.5	
7	3.0	
8	3.5	
9	4.0	
10	4.5	
11	5.0	
12	5.5	
13	6.0	

### Nature of Graph



### CALCULATION:

Normality of NaOH = 0.1N

Volume of NaOH required to neutralize HCl in acid Mix. = \_\_\_\_\_  $\text{cm}^3$  ( $V_1$ )

$$\begin{aligned} \text{Normality of HCl} &= \frac{N_{\text{NaOH}} \times V_{\text{NaOH}}}{V_{\text{Acid Mixture}}} \\ &= \frac{0.1 \times V_1}{50} \end{aligned}$$

Therefore Amount of HCl in acid mixture =  $N_{\text{HCl}} \times 36.5 \text{ gm/lit}$   
= .....gm/lit

Volume of NaOH required to neutralize both HCl  
&  $\text{CH}_3\text{COOH}$  in acid mixture = -----  $\text{cm}^3$  ( $V_2$ )

Volume of NaOH required to neutralize  $\text{CH}_3\text{COOH}$  IN Acid mixture =  $v_2 - \text{-----} \text{cm}^3$   $v_1$

$$\begin{aligned} \text{Normality of } \text{CH}_3\text{COOH} &= \frac{N_{\text{NaOH}} \times V_{\text{NaOH}}}{V_{\text{Acid Mixture}}} \\ &= \frac{0.1 \times (V_2 - V_1)}{50} \end{aligned}$$

Therefore Amount of  $\text{CH}_3\text{COOH}$  in acid mixture =  $N_{\text{CH}_3\text{COOH}} \times 60 \text{ gm/lit}$   
= .....gm/lit





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### **EXPERIMENT NO.03**

#### **3. DETERMINATION OF VISCOSITY COEFFICIENT OF A GIVEN LIQUID USING OSTWALD'S VISCOMETER**

##### **AIM :**

To determine the viscosity coefficient of the given liquid by using ostwald's viscometer.

##### **PRINCIPLE :**

Viscosity of liquid may be defined as the resistance that one part of a fluid offers to the flow of another part of the liquid. The coefficient of viscosity ( $\eta$ ) is defined as the force per unit area required to move a layer of fluid with a unit velocity difference past another parallel layer at unit distance away. In cgs system of units, the viscosity coefficient of a fluid is expressed in poises.

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

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

where  $\eta_1$  and  $\eta_2$  = viscosity coefficient of liquids.

$d_1$  and  $d_2$  equal to density of liquids.

$t_1$  and  $t_2$  equal to time taken to flow through capillary tube (liquid)

The flow times for the liquid and water are determined in a Ostwald's viscometer (Fig) Knowing the densities of the liquid and water and the viscosity coefficient of water. Viscosity coefficient of liquid can be calculated.

##### **PROCEDURE :**

Take a clean and dry viscometer and fix it vertically to a stand in water taken in a jar. In such a way that the mark above the upper bulb is below the water level. Pipette out a defined known volume (10ml or 20ml depending upon the size of the upper bulb) of the liquid into the lower bulb. With the help of a rubber tubing fitted to the limb containing the upper bulb. Suck the liquid into the upper bulb and above the mark A of the viscometer. Allow it to flow freely through the capillary. When the level of the liquid just crosses the mark A, start a stop clock. Stop the clock when the liquid crosses the lower mark B. note the flow time in seconds. Repeat for two more time.

Remove the liquid from the viscometer, rinse with acetone and dry it. Now pipette out the same volume of distilled water as before into the lower bulb of the viscometer and find out the flow time as before.

##### **RESULT :**

Viscosity coefficient of the given liquid = ..... centipoises.



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### OBSERVATION AND CALCULATION :

Density of water  $d_1 = \dots\dots\dots \text{gm/cc}$   
 Viscosity coefficient of water  $\eta_1 = \dots\dots\dots \text{Centipoise}$   
 Density of the liquid  $d_2 = \dots\dots\dots \text{gm/cc}$

### To determine the flow time

	Time of flow (seconds)			
	Trial – I	Trial – II	Trial – III	Average
Liquid				
Water				

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

∴ Viscosity coefficient the liquid.  $\eta_2 = \frac{t_2 d_2 \times \eta_1}{t_1 d_1}$

= ..... centipoises.



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### EXPERIMENT NO.04

#### 4. ESTIMATION OF COPPER COLORIMETRICALLY.

##### PRINCIPLE:

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

$$I_o = I_a + I_r + I_t.$$

For a glass – air interface  $I_r$  is negligible, therefore,

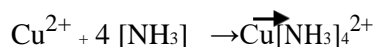
$$I_o = I_a + I_t$$

$I_t / I_o = T$  called the transmittance,  $\log I_o / I_t$  is called the absorbance or optical density  $A$ . The relation between absorbance  $A$ , concentration  $c$  (expressed in mol/lit) and path length (expressed in cm) is given by Beer = Lambert's law.

$$A = \log I_o / I_t = \sum_{ct}.$$

Where  $\sum$  is the molar extinction coefficient, a constant for a given substance at a given wavelength, if the path length (cell thickness) is kept constant, then,  $A$  is proportional to  $C$ . Hence a plot of absorbance against concentration gives a straight line as shown in figure (calibration curve). The optical density is measured using a spectrophotometer at a wavelength where the absorbance is maximum.

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



The absorbance of each of these solutions is measured at 620 nm since the complex shown maximum absorbance at this wavelength. The concentration is plotted against absorbance to get a calibration curve. A known volume of the test solution is treated with strong ammonia and diluted to the same volume as above. The absorbance of this solution at 620 nm is measured and its concentration is determined from the calibration curve.

##### PROCEDURE:

Place the given copper sulphate solution (0.02M stock solution) in a burette and transfer 2 ml, 4 ml, 6 ml, 8 ml and 10 ml of the solution into separate labeled 25 ml standard volumetric flask. Add 1 ml of ammonia solution to each of them and make up to the mark with ion exchange water (distilled water) stopper the flasks and mix the solutions well. To the test solution taken in a 25 ml standard volumetric flask, add 1 ml of Ammonia solution and make up the mark. Mix well, prepare a blank solution by diluting 1 ml of Ammonia solution in a 25 standard volumetric flask, make up to the mark with distilled water and mix well. First adjust 0.00 absorbance on Photoelectric Colorimeter with blank solution. After

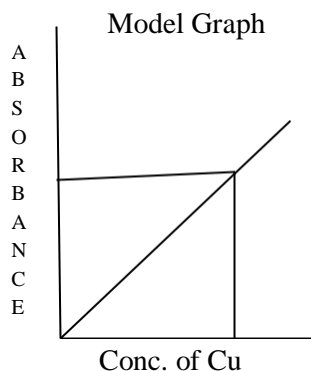


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10 minutes, measure the absorbance of the solutions against blank at 620 nm using a photoelectric colorimeter. Tabulate the readings as shown. Draw a calibration curve by plotting concentration of copper against absorbance. Using the calibration curve determine the concentration of copper in the test solution and calculate the amount of copper in the solution.

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



**Tabular column**

Volume of $\text{Cm}^3$	Conc. of Copper	Absorbance
2		
4		
6		
8		
10		
Test solution		

### **CALCULATION:**

Concentration of given known Conc<sup>n</sup> copper solution = 0.02M

Concentration of 2ml labelled prepared 25ml copper solution =  $\frac{2 \times 0.02}{25} = 1.6 \times 10^{-3}$

(Similarly calculate Conc<sup>n</sup> 4ml, 6ml, 8ml, 10ml prepared labeled Solution)

### **Result :**

- (i) Concentration of Cu in given test solution from graph = ..... M
- (ii) Amount of Cu in given test solution = Conc of Cu x 63.54 x 25mg



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### EXPERIMENT NO.05

#### 5.DETERMINATION OF pKa OF WEAK ACID USING pH METER.

##### PRINCIPLE:

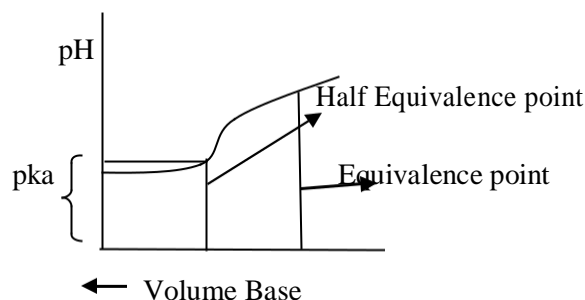
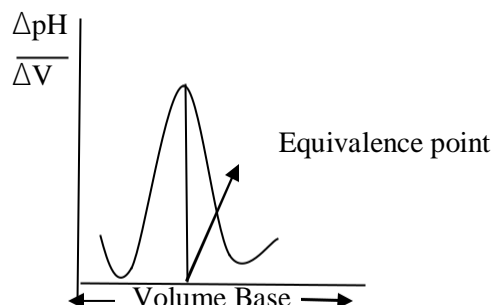
In the titration of an acid with a base, the pH of the solution rises gradually at first, then more rapidly, until at the equivalence point, there is a very sharp increase in pH for a, very small quantity of added base. Beyond the equivalence point, the pH increase only slightly on addition of excess base. The titration curve obtained by plotting changes in pH at different amount of the base added. pka is a measure of the dissociation constant ( $K_a$ ) of an acid. The pka value of an acid can be calculated using Henderson – Hasselbalch equation.

$$\text{pH} = \text{pka} + \log [\text{salt}] / [\text{acid}]$$

Where  $\text{pka} = -\log K_a$ .

At half equivalence point,  $[\text{salt}] = [\text{acid}]$  and hence  $\text{pH} = \text{pka}$ . Thus pH at half equivalence point gives the pka value of weak acid.

##### MODEL GRAPH



##### PROCEDURE:-

Transfer 50 ml of the given weak acid (acetic acid) into a beaker using a pipette,. Immerse a combined glass electrode assembly into the acid and connect the cell to a pH meter. Measure the pH of the acid Fill a burette with the base (Sodium Hydroxide) stir the solution carefully and measure the pH after each addition of 0.5ml. When the pH begins to a tendency to increase rapidly. Than add small increments (say 0.5ml) of the base and measure the pH after each addition. Till there is a slight increase in pH on the addition of the base. Tabulate the reading as given below.



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**OBSERVATION**

Volume of acid sample	Volume of base added NaOH (0.1N)	pH	$\Delta\text{pH}$	$\Delta V$	$\Delta\text{pH} / \Delta V$
50ml acid sample	0.0		--	--	--
	0.5			0.5	
	1.0			0.5	
	1.5			0.5	
	2.0			0.5	
	2.5			0.5	
	3.0			0.5	
	3.5			0.5	
	4.0			0.5	
	4.5			0.5	
	5.0			0.5	
	5.5			0.5	
	6.0			0.5	
	6.5			0.5	
	7.0			0.5	
	7.5			0.5	

Plot a graph of  $\Delta\text{pH} / \Delta V$  against Volume of NaOH and determine the equivalence point, Plot a graph of pH (ordinate) against the volume of sodium hydroxide added (abscissa). Determine the equivalence point and hence the pH at half the equivalence point. This gives the pKa of the acid.

**Result :** The pKa value of weak acid \_\_\_\_\_



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### EXPERIMENT NO.06

#### 6. ESTIMATION OF TOTAL HARDNESS OF A WATER EDTA COMPLEXOMETRIC METHOD.

##### AIM:

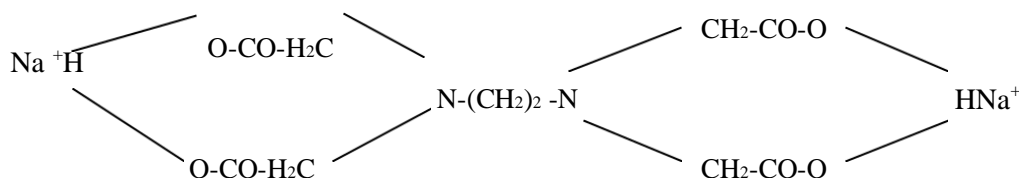
To estimate volumetrically the total hardness of water by using standard Ethylene Diamine Tetra Acetic acid (EDTA).

**APPARATUS:** Burette, pipette, conical flask, Beaker

##### PRINCIPLE:

Hardness in water is that characteristic which prevents the lathering of soap. This is due to the presence of certain salts of calcium and magnesium ions. The hardness is classified as Temporary and Permanent hardness. Temporary hardness, which is due to presence of  $\text{Ca}(\text{HCO}_3)_2$  can be removed by boiling permanent hardness, which is due to the presence of chlorides, sulphates of calcium and magnesium is not destroyed by boiling.

In this complex metric method of determination of hardness of water. Dio-sodium salt of EDTA will be used.



##### DISODIUM SALT OF EDTA

- Hardness producing constituent can be estimated by titration with EDTA salts solution in the presence of Eriochrome Black – T (EBT) indicator.
- EBT Indicator, when added in small amount to hard water, Buffered to a pH of 10, combines with a few  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  ions to form wine-red unstable complex.  

$$\text{Ca}^{2+} \text{ or } \text{Mg}^{2+} + \text{EBT indicator} \longrightarrow \text{-(Ca-EBT)unstable complex (wine Red Colour)}$$

or (Mg- EBT)
- During titration with EDTA free metal ions combines with EDTA and forms colorless stable complex.  

$$\text{EDTA} + \text{free Ca}^{2+} \text{ or } \text{Mg}^{2+} \longrightarrow \text{Ca or EDTA – Mg (stable Complex)}$$
- When free metal ions are not available, EDTA extracts the metal ion from the metal ion-indicator complex there by releasing the free indicator.  

$$\text{EDTA} + \text{-(Ca-EBT) or (Mg- EBT)} \longrightarrow \text{EDTA -Ca + free EBT (Blue in color)}$$

**End point.**

##### Determination of Total Hardness.

##### PROCEDURE:

First rinse & fill the burette with 0.01M standard EDTA solution. Now pipette 50ml Hardwater sample in conical flask, add 2ml pH10 buffer solution & pinch of EBT indicator. The colour of the solution becomes wine red which is then titrated with standard EDTA solution by adding drop wise EDTA solution from the burette till the colour changes from wine red to clear blue. The final reading is noted and from the titre value total Hardness of water is determined.



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**Observation:**

In burette : ..... standard EDTA solution

$M_{\text{EDTA}} = \dots\dots\dots M$

**Conical flask** : 50 ml tap water + 1-2ml pH-10 Buffer solution + pinch of EBT indicator.

**Indicator** : Erio Chrome Black-T Indicator

**Colour Changes** : Wine Red colour changes to blue colour, at the end point

**TABULAR COLUMN**

Sl.No.	Volume of water Pipetted	Burette Reading		Volume of EDTA Sol'n Consumed (b-a)	Concordant Reading
Pilot		Intital (a)	Final (b)		
I					
II					
III					

**CALCULATION:**

$$\text{Total Hardness of water} = \frac{M_{\text{EDTA}} \times V_{\text{EDTA}}}{V_{\text{WATER}}} \times \frac{1000}{0.01}$$

(since 1 ml of 0.01 m EDTA = 1mg CaCO<sub>3</sub> eqt. Hardness)

**Result :**

Total hardness of water = ..... ppm (Parts Per Million)





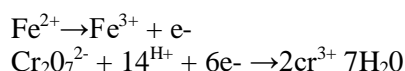
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## **DEPARTMENT OF APPLIED SCIENCES & HUMANITIES**

### **EXPERIMENT NO.07**

#### **7. ESTIMATION OF IRON IN TMT BAR BY DIPHENYLE AMINE**

**Principle:** The iron present in the TMT bar can be estimated by first taking a known weight of sample and then it is dissolved in 1:1 H<sub>2</sub>SO<sub>4</sub> heating. The sample solution is then cooled and diluted up to mark in 100ml volumetric flask. The iron II solution is then estimated by titrating quickly against standard K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution using diphenyle amine indicator till the colour changes from green to blue-violet. From the volume of standard K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> consumed. The iron present in the TMT bar is estimated.



#### **Procedure:**

I) Preparation of TMT bar solution → Weight out 0.1 gram of T.M.T bar sample and add 20 ml 1:1 dil H<sub>2</sub>SO<sub>4</sub> and heat it gently till it dissolves. It is then cooled and made up to mark by distilled water in 100ml volumetric flask

II) Determination of iron in sample solution pipette out 20 ml of Fe<sup>2+</sup> solution in conical flask. To this add 1 test tube dil H<sub>2</sub>SO<sub>4</sub> and 2-3 ml of 1:1 Phosphoric acid followed by 3-4 drops of diphenyle amine indicator. Now titrate the solution with standard K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution till the colour changes from green to blue violet at the end point.

#### **OBSERVATION :**

**In burette** = ...0.05 N K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution

**In Conical flask** : 20ml Fe<sup>2+</sup> solution + 1 test tube dil H<sub>2</sub>SO<sub>4</sub> + 2-3 ml (1:1) phosphoric acid + 3-4 drops of diphenyle amine indicator.

**Titrate** : till the colour changes from green to blue violet at the end point.

**Indicator** : diphenyle amine indicator

**End Point** : (colour changes) green to blue violet



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(TABULAR COLUMN)

Sl.No.	Volume of Iron Solution	Burette Reading		Volume of $K_2Cr_2O_7$	Concordant
Pilot	Pipetted	Intitial (a)	Final (b)	Sol'n Consumed (b-a)	Reading
I					
II					
III					

**CALCULATION:**

$$\text{Normality of iron (II) solution} = \frac{V_{K_2Cr_2O_7} \times N_{K_2Cr_2O_7}}{V_{\text{Iron II Solution}}}$$

$$= \underline{\text{'x'}}. N$$

$$\begin{aligned} \text{Amount of iron (II) 1000ML of sample solution} &= N_{\text{iron II SOLUTION}} \times 55.85 \\ &= \text{'x'} \times 55.85 \text{ gram/liter} \\ &= \text{'A'} \text{ gram/liter} \end{aligned}$$

$$\% \text{ Iron II in T.M.T bar} = \frac{A \times 100}{\text{Weight of iron taken}}$$



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## DEPARTMENT OF APPLIED SCIENCES & HUMANITIES

### EXPERIMENT NO.08

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

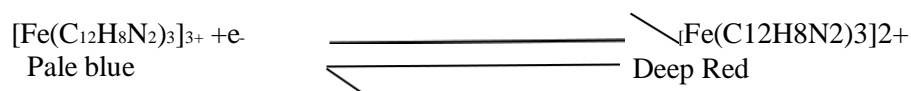
**AIM:** To estimate the chemical oxygen demand of water.

#### PRINCIPLE :

Chemical oxygen demand (COD) parameter has been introduced to measure the total oxidisable impurities present in the Sewage. This includes both, biologically oxidisable and biologically inert but chemically oxidisable impurities in the water. It is the amount of oxygen equivalent used while oxidizing the chemically oxidisable impurities present in water, with a strong chemical oxidant,  $K_2CrO_7$  in acid medium. It is expressed in  $mg. dm^{-3}$  or ppm (parts per million).

A known volume of the waste water sample is refluxed with excess of Potassium dichromate solution in sulphuric acid medium and in the presence of  $AgSO_4$  and  $HgSO_4$ . Potassium dichromate oxidizes all oxidisable impurities.  $AgSO_4$  catalyses the oxidation of straight chain organic compounds, aromatic and pyridine.  $HgSO_4$  avoids the interference of  $Cl^-$  ions precipitates silver ions as  $AgCl$ . The amount of unreacted potassium dichromate is determined by titration with standard Mohr's salt solution. The amount of potassium dichromate solution consumed corresponds to the COD of the water sample. To calculate the COD, a blank titration without the water sample is carried out.

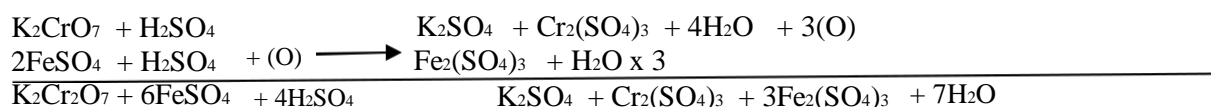
Ferroin (1:10 phenanthraline – iron (II) complex) is used as the indicator in the titration to detect the end point. The indicator is intensely red in color. Strong oxidizing agents oxidize the indicator to iron (III) complex, which has a pale blue color.



As long as  $K_2CrO_7$  solution is present in the solution the indicator is in the oxidized form, imparting blue color to the solution. The solution becomes blue green as the titration continues due to the formation of  $Cr(III)$  ions as a result of reduction of  $CrO_7^{2-}$  i.e. when  $K_2CrO_7$  is completely exhausted in the solution, the oxidized form of the indicator gets reduced to the reduced form, imparting red-brown color to the solution. Therefore, the end point is marked by the color change from blue-brown. The end point is sharp only at high acid concentration and therefore the solution is kept at a very high acidity of  $H_2SO_4$ .

A standard solution of Mohr's salt is prepared by dissolving a known weight of the salt in a known volume of the solution. A test tube of dil Sulphuric acid is added during dissolution to prevent the hydrolysis of the salt in the solution.

The reaction between Mohr's salt and  $K_2CrO_7$  can be represented as follows.





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#### Part : A (Determination of COD)

Pipette out 20 ml of the waste water sample into 250ml conical flask with a ground glass neck. Add 1 gram of  $\text{HgSO}_4$  followed by 2 test tubes of Silver Sulphate / Sulphuric Acid solution (prepared by dissolving about 5 grams of  $\text{AgSO}_4$  in 250 ml of Conc.  $\text{H}_2\text{SO}_4$  and then slowly diluting to 500 ml with distilled water). Then pipette out 20 ml of 0.05N  $\text{K}_2\text{CrO}_7$  solution into the flask. Fit the flask with the reflux condenser and reflux the mixture for about half an hour. On cooling, rinse the inside of the

condenser with distilled water into the flask. Add 2-3 drops of ferroin Indicator and titrate against standard Mohr's Salt solution till the color changes from blue green to reddish brown.

#### Part : B (Blank Titration)

Repeat the above procedure by taking 20 ml distilled water in the place of sample waste water.

#### OBSERVATION & CALCULATION

**Burette Solution** : A Standard Mohr's Salt Solution

**Conical Flask** : 20ml Waste water Sample + 5 ml Mixed solution  $\text{AgSO}_4$  and  $\text{HgSO}_4$  in Conc.  $\text{H}_2\text{SO}_4$

→ solution + 10ml 0.05N Potassium Dichromate Solution + 1 test tube Dil.  $\text{H}_2\text{SO}_4$  boil for 2 minute

→ Cool → then add Ferroin Indicator (2 to 3 drops)

**Color Change** : Blue Green to Reddish Brown at end point.

#### TABULAR COLUMN

Sl.No.	Volume of waste water sample Pipetted	Burette Reading		Volume of Mohr's Salt solution Consumed (b-a)	Concordant Reading
Pilot		Initial (a)	Final (b)		
I					
II					
III					



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**Calculation :**

Normality of Standard Mohr's salt solution = 'X' N.

Volume of Mohr's Salt consumed by excess of Potassium Dichromate after oxidizing impurities in 20ml water sample =  $V_1$  .....ml

Volume of Mohr's Salt consumed in blank titration : =  $V_2$ .....ml

Volume of Potassium Dichromate consumed by Oxidisable impurities in terms of volume of Mohr's Salt solution =  $V_2 - V_1$

1000 ml of 1 N Mohr's Salt Solution = 8 grams of Oxygen

$$\begin{aligned} \text{COD of the sample (per liter)} &= \frac{(V_{\text{FAS for blank}} - V_{\text{FAS for sample}}) \times N_{\text{FAS}}}{V \text{ waste water sample}} \times 8 \times 1000 \\ &= \text{..... ppm (Mg.dm}^{-3}\text{)} \end{aligned}$$

Result:

The COD of given waste water sample = ..... ppm (Mg.dm<sup>-3</sup>)



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#### **EXPERIMENT NO .09**

### **9. SYNTHESIS OF IRON OXIDE NANOPARTICLES BY PRECIPITATION METHOD**

**AIM:** Synthesis of Iron oxide Nanoparticles by precipitation method

#### **CHEMICAL REQUIRED :**

Ferrous sulphate solution (0.2) and Ferric chloride solution (0.1M), Deionized water and ammonia (3M)

#### **PRINCIPLE:**

Co-precipitation is very convenient way to synthesize Iron oxide nanoparticles ( $\text{Fe}_3\text{O}_4$ ) from aqueous  $\text{Fe}^{2+}$  /  $\text{Fe}^{3+}$  salt solution by addition of base (Ammonia) at room temperature or at elevated temperature. The black precipitate of  $\text{Fe}_3\text{O}_4$  is obtained which is filtered, dried and characterized



#### **PROCEDURE:**

- i) Dissolve 0.4 gram of  $\text{FeSO}_4 \cdot 2\text{H}_2\text{O}$  and 1.4 gram of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  in 50ml deionised water.
- ii) Add ammonia solution (3M) with constant stirring at 1000 rpm to the above solution till pH=10 is obtained
- iii) The result is formation of black precipitate of  $\text{Fe}_3\text{O}_4$  which is then filtered and dried in oven at  $200^\circ\text{C}$  for two hours.
- iv) The obtained product is  $\text{Fe}_3\text{O}_4$  nanoparticles with magnetic properties

#### **INTERPRETATION OF RESULT**

Characterization of magnetic  $\text{Fe}_3\text{O}_4$  nanoparticles

- i) UV visible spectroscopy is used to prove the existence of nanoparticle
- ii) The morphology and size is determined by transmission electron microscopy (TEM) scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis.



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#### **EXPERIMENT NO.10**

### **10. DETERMINATION OF STRENGTH OF ACID IN LEAD-ACID BATTERY**

**AIM:** Determination of strength of Acid in lead-Acid battery

**APPARATUS:** Hydrometer

#### **PRINCIPLE:**

Lead-Acid battery is filled with electrolyte  $H_2SO_4$  (20-30%) with specific gravity ranging for 1.2 to 1.22 at temperature of  $25^\circ C$  normally Sp.Gr of lead-acid battery slightly increases for first 2 years and then it starts gradually decreasing till the end of life.

Hydrometer is used to measure Sp.Gr of electrolyte (Acid) in lead-acid battery and its healthiness condition. Hydrometer is a device which possesses sealed glass bulb, The neck of the glass bulb is calibrated to read specific gravity the sample of electrolyte (Acid) is collected by squeezing the bulb of hydrometer and Sp.Gr of electrolyte in the battery can be measured by looking at scale of hydrometer up to which liquid level is raised, Three different colours are marked to check the healthiness condition of electrolyte in battery. Green colour indicates that the battery is fully charged and yellow colour indicates electrolyte level is low and battery need to charge whereas red colour indicates that battery is dead and too old to use.

Reading of Sp.Gr of Acid on the scale of hydrometer	<b>Sp.Gr</b>	<b>State of charge</b>
	1.277	100%
	1.258	90%
	1.238	70%
	1.195	60%
	1.172	50%
	1.148	40%
	1.124	30%
	1.098	20%
	1.073	10%
	1.000	0%

#### **PROCEDURE:**

Rubber bulb of Hydrometer should be squeezed several times before taking electrolyte (Acid) sample from the battery. For taking electrolyte from the battery, bulb is released and sample can be collected. Sp.Gr of electrolyte in the battery can be measured by looking at scale of hydrometer up to which liquid level is raised from the Sp.Gr of the electrolyte for different cell compartment of the battery the healthiness and charging ability of battery is measured from the table discussed above in the principle.



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