**K.S. SCHOOL OF ENGINEERING & MANAGEMENT**
**No. 15, Mallasandra, Off. Kanakapura Road,**
**Bangalore – 560 062**

# APPLIED CHEMISTRY LABORATORY MANUAL

**For**

**(BCHES102/202, BCHEE102/202, BCHEC102/202, BCHEM102/202 Streams)**

**(First/Second Semester B.E)**

**Academic Year 2023 – 2024**

**Name of the Student :** ____________________

**Branch/Section :** ____________________

**USN No. :** ____________________

**Batch No. :** ____________________

# CIE Assessment Details

| Continuous Internal Evaluation (CIE) | Total |
|---|---|
| ➢ Conduction of each experiment with results **:** 5 Marks<br>➢ Brief Procedure write up**:** 5 marks<br>➢ Record (Neatness and on time submission) **:** 5 Marks<br>➢ Lab test: 10 Marks (Conducted for 50 marks and reduced to 10 marks) | **25 Marks** |
| **Note:** In SEE, the questions from the laboratory component shall be included**.** The maximum of 05 questions is to be set from the practical component, the total marks of all questions should not be more than 25 marks. | |

# General Instructions

1. Keep your working table always clean & neat.
2. Do not contaminate the reagents & always keep the reagent bottles in their proper places. Do not alter their order.
3. Keep water taps and gas taps closed when not in use.
4. Do not throw any waste paper into the sink.
5. Do not pour concentrated acids directly into the sink. If they are to be poured, flush them using water liberally.
6. To turn out to be a better analyst, understand the theory behind the reactions you carry out.
7. Record your observations as and when you proceed and not after completion. Have a separate notebook for recording and keep it away from the reagents and sink.
8. Use the specified quantities of reagents.
9. Do not use wet hand during weighing.

## DO’S

- MAINTAIN OBSERVATION BOOK.
- CARRY GRAPH SHEETS, CALCULATOR, PENCIL AND ERASER TO LAB.
- WASH ALL THE GLASSWARE AND HANDOVER TO THE LAB INSTRUCTOR.
- SWITCH OFF THE INSTRUMENT AFTER THE EXPERIMENT IS OVER.
- REPORT THE BREAKAGE IMMEDIATELY.
- MAINTAIN SCILENCE INSIDE LAB.

## DONT’S

- DO NOT OPERATE INSTRUMENTS UNLESS YOU ARE INSTRUCTED.
- DO NOT WASTE DISTILLED WATER.
- DO NOT WASTE CHEMICALS.
- DO NOT LEAVE THE LAB WITHOUT PERMISSION.
- DO NOT DISCARD THE ACID TO THE SINK DIRECTLY.

# APPLIED CHEMISTRY PRACTICAL MODULE

# For

# Computer Science and Engineering and Allied branches (BCHES102/202)

***__A – Demonstration (any two) offline/virtual:__***

A1. Chemical Structure drawing using software: ChemDraw or ACD/ChemSketch
A2. Determination of strength of an acid in Pb-acid battery
A3: Synthesis of Iron-oxide Nanoparticles
A4. Electrolysis of water

***__B – Exercise (compulsorily any 4 to be conducted):__***

B1. Conductometric estimation of acid mixture
B2. Potentiometric estimation of FAS using $K_2Cr_2O_7$
B3. Determination of pKa of vinegar using pH sensor (Glass electrode)
B4. Determination of rate of corrosion of mild steel by weight loss method
B5. Estimation of total hardness of water by EDTA method

***__C – Structured Enquiry (compulsorily any 4 to be conducted):__***

C1. Estimation of Copper present in electroplating effluent by optical sensor (colorimetry)
C2. Determination of Viscosity coefficient of lubricant (Ostwald's viscometer)
C3. Estimation of iron in TMT bar by diphenyl amine/external indicator method
C4. Estimation of Sodium present in soil/effluent sample using flame photometry
C5. Determination of Chemical Oxygen Demand (COD) of industrial waste water sample

***__D – Open Ended Experiments (any two):__***

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

# APPLIED CHEMISTRY PRACTICAL MODULE

## For

## Electrical and Electronics Engineering stream (BCHEE102/202)

***A – Demonstration (any two) offline/virtual:***

A1. Synthesis of polyurethane

A2. Determination of strength of an acid in Pb-acid battery

A3. Synthesis of iron oxide nanoparticles

A4. Electroplating of copper on metallic objects

***B – Exercise (compulsorily any 4 to be conducted):***

B1. Conductometric estimation of acid mixture

B2. Potentiometric estimation of FAS using $K_2Cr_2O_7$

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

B4. Determination of rate of corrosion of mild steel by weight loss method

B5.Estimation of total hardness of water by EDTA method

***C – Structured Enquiry (compulsorily any 4 to be conducted):***

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

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

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

C4. Estimation of Sodium present in soil/effluent sample using flame photometry

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

***D – Open Ended Experiments (any two):***

D1. Estimation of metal in e-waste by optical sensors

D2. Electroless plating of Nickle on Copper

D3. Determination of glucose by electrochemical sensors

D4. Synthesis of polyaniline and its conductivity measurement

# APPLIED CHEMISTRY PRACTICAL MODULE

# For

## Civil Engineering stream (BCHEC102/202)

***A – Demonstration (any two) offline/virtual:***

A1. Synthesis of polyurethane
A2. Quantitative estimation of Aluminium by precipitation Method
A3. Synthesis of iron oxide nanoparticles
A4. Determination of chloride content in the given water sample by Argentometric method

***B – Exercise (compulsorily any 4 to be conducted):***

B1. Conductometric estimation of acid mixture
B2. Potentiometric estimation of FAS using $K_2Cr_2O_7$
B3. Determination of pKa of vinegar using pH sensor (Glass electrode)
B4. Determination of rate of corrosion of mild steel by weight loss method
B5.Estimation of total hardness of water by EDTA method

***C – Structured Enquiry (compulsorily any 4 to be conducted):***

C1. Estimation of Copper present in electroplating effluent by optical sensor (colorimetry)
C2. Determination of Viscosity coefficient of lubricant (Ostwald's viscometer)
C3. Estimation of iron in TMT bar by diphenyl amine/external indicator method
C4. Estimation of Sodium present in soil/effluent sample using flame photometry
C5. Determination of Chemical Oxygen Demand(COD) of industrial waste water sample

***D – Open Ended Experiments (any two):***

D1. Gravimetric estimation of gypsum in Portland cement
D2. Electroplating of desired metal on substrate
D3. Estimation of manganese dioxide in pyrolusite
D4. Analysis of cement for its components

# APPLIED CHEMISTRY PRACTICAL MODULE

# For

## Mechanical Engineering stream (BCHEM102/202)

***A – Demonstration (any two) offline/virtual:***

A1. Synthesis of polyurethane
A2. Preparation of urea formaldehyde resin
A3. Synthesis of iron oxide nanoparticles
A4. Determination of acid value of biofuel

***B – Exercise (compulsorily any 4 to be conducted):***

B1. Conductometric estimation of acid mixture
B2. Potentiometric estimation of FAS using $K_2Cr_2O_7$
B3. Determination of pKa of vinegar using pH sensor (Glass electrode)
B4. Determination of rate of corrosion of mild steel by weight loss method
B5. Estimation of total hardness of water by EDTA method

***C – Structured Enquiry (compulsorily any 4 to be conducted):***

C1. Estimation of Copper present in electroplating effluent by optical sensor (colorimetry)
C2. Determination of Viscosity coefficient of lubricant (Ostwald's viscometer)
C3. Estimation of iron in TMT bar by diphenyl amine/external indicator method
C4. Estimation of Sodium present in soil/effluent sample using flame photometry
C5. Determination of Chemical Oxygen Demand (COD) of industrial waste water sample

***D – Open Ended Experiments (any two):***

D1. Estimation of percentage of iron in steel
D2. Electroplating of desired metal on substrate
D3. Synthesis of biodiesel
D4. Synthesis of Aluminium Oxide nano particle

# CONTENTS

## DEMONSTRATION EXPERIMENT - A1

## SYNTHESIS OF IRON – OXIDE NANOPARTICLES

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

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

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

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

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

**Reaction:**

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

**Report:** Weight of Iron Oxide nanoparticles = -------------g

## DEMONSTRATION EXPERIMENT - A2

## ELECTROLYSIS OF WATER

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

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

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

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

The reactions taking place at cathode and anode of the cell during electrolysis of water in an acid medium are given below.

Cathode (reduction): $4\ H_2O(l) + 4e^- \rightarrow 2H_2(g) + 4\ OH^-(aq)$

Anode (oxidation): $4\ OH^-(aq) \rightarrow O_2(g) + 2H_2O(l) + 4\ e^-$

Overall reaction: $2\ H_2O(l) \rightarrow 2\ H_2(g) + O_2(g)$

## DEMONSTRATION EXPERIMENT - A3

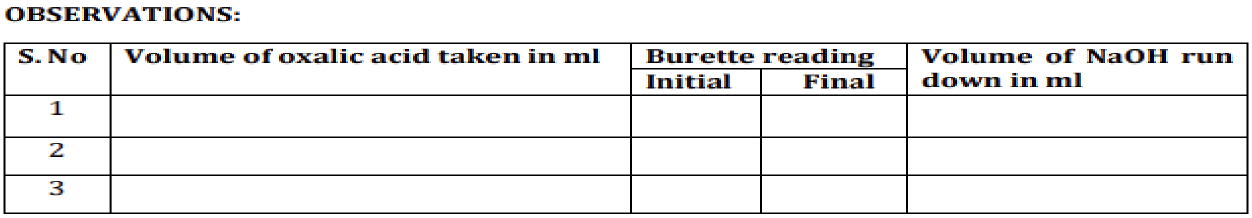
# DETERMINATION OF STRENGTH OF AN ACID IN Pb-ACID BATTERY

## Observation and Calculations

### STEP-1: Preparation of standard oxalic acid solution

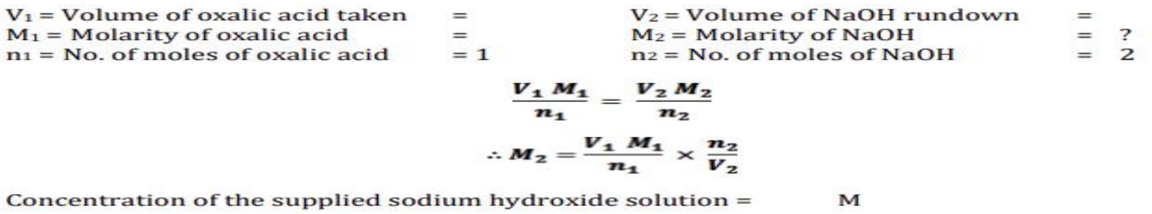
0.025M Oxalic acid primary standard solution is prepared by dissolving 0.315g of Oxalic acid solid in 100 ml distilled water.

### STEP-2: Standardization of sodium hydroxide solution



**OBSERVATIONS:**

| S. No | Volume of oxalic acid taken in ml | Burette reading | | Volume of NaOH run down in ml |
|---|---|---|---|---|
| | | Initial | Final | |
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |



$V_1$ = Volume of oxalic acid taken =
$M_1$ = Molarity of oxalic acid =
$n_1$ = No. of moles of oxalic acid = 1

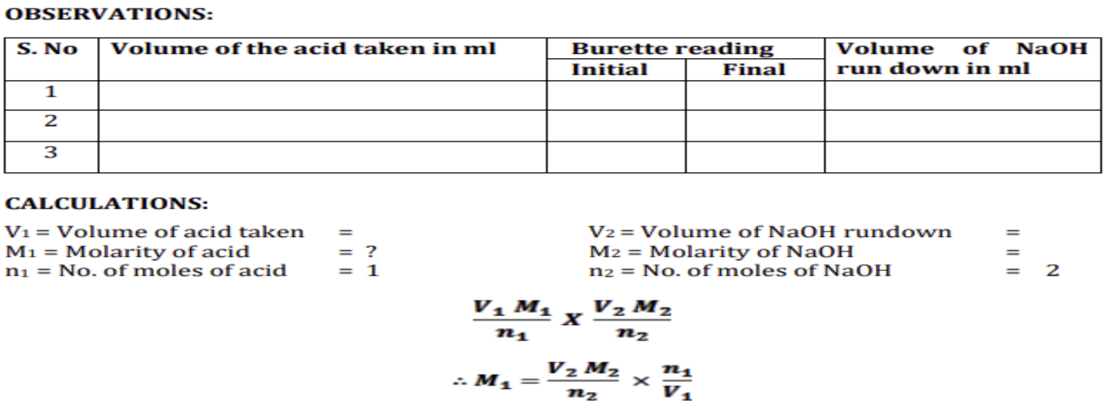
$V_2$ = Volume of NaOH rundown =
$M_2$ = Molarity of NaOH = ?
$n_2$ = No. of moles of NaOH = 2

$$\frac{V_1 M_1}{n_1} = \frac{V_2 M_2}{n_2}$$

$$\therefore M_2 = \frac{V_1 M_1}{n_1} \times \frac{n_2}{V_2}$$

Concentration of the supplied sodium hydroxide solution = M

### STEP-3: Determination of acid strength of lead acid battery:



**OBSERVATIONS:**

| S. No | Volume of the acid taken in ml | Burette reading | | Volume of NaOH run down in ml |
|---|---|---|---|---|
| | | Initial | Final | |
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |

**CALCULATIONS:**

$V_1$ = Volume of acid taken =
$M_1$ = Molarity of acid = ?
$n_1$ = No. of moles of acid = 1

$V_2$ = Volume of NaOH rundown =
$M_2$ = Molarity of NaOH =
$n_2$ = No. of moles of NaOH = 2

$$\frac{V_1 M_1}{n_1} x \frac{V_2 M_2}{n_2}$$

$$\therefore M_1 = \frac{V_2 M_2}{n_2} \times \frac{n_1}{V_1}$$

Concentration of the supplied test sample solution = M

## DEMONSTRATION EXPERIMENT - A3

# DETERMINATION OF STRENGTH OF AN ACID IN Pb-ACID BATTERY

### THEORY/PRINCIPLE

A storage cell is that which can work both as voltaic cell as well as electrical cell. During its operation it works as a voltaic cell i.e., it supplies electrical energy due to a chemical energy and it becomes discharged. Then during its charging, it works as an electrolytic cell i.e., chemical is produced by supplying of electrical energy. It is also called as lead storage battery.
Anode : Lead (Pb)
Cathode : Lead dioxide ($PbO_2$)
Electrolyte : Dil. $H_2SO_4$
A lead storage battery consists of 6 cells each producing 2V output. To increase the current output of each cell, the cathode plates are joined together and the anode plates are also joined together and connected in series, we get an output of 12V as shown in figure.

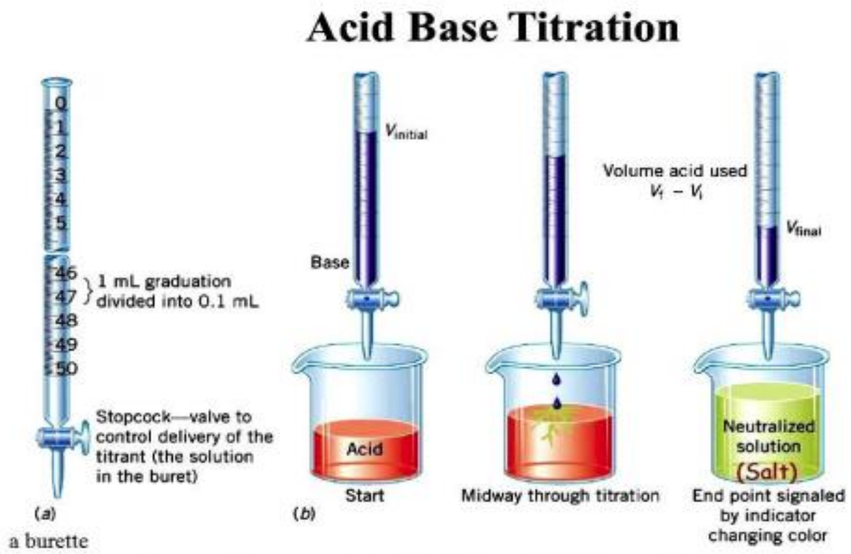
**Electrode reactions during discharge**

Anode : $Pb \longrightarrow Pb^{2+} + 2e^-$
$Pb^{2+} + SO_4^{2-} \longrightarrow PbSO_4$
Cathode : $PbO_2 + 4H^+ + 2e^- \longrightarrow Pb^{2+} + 2H_2O$
$Pb^{2+} + SO_4^{2-} \longrightarrow PbSO_4$
Net reaction : $Pb + PbO_2 + 4H^+ + 2SO_4^{2-} \longrightarrow 2PbSO_4 + 2H_2O$

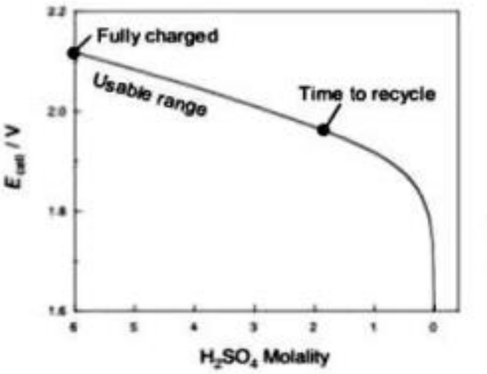
**Electrode reactions during charging**

Anode : $PbSO_4 + 2e^- \longrightarrow Pb^{2+} + SO_4^{2-}$
Cathode : $PbSO_4 + 2H_2O \longrightarrow PbO_2 + SO_4^{2-} + 4H^+ + 2e^-$
Net reaction : $2PbSO_4 + 2H_2O \longrightarrow Pb + PbO_2 + 4H^+ + 2SO_4^{2-}$

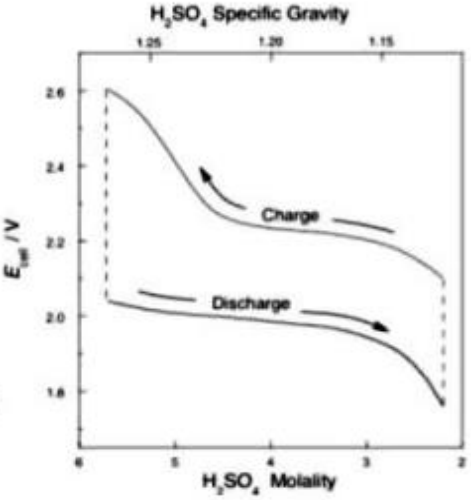
The $PbSO_4$ forms during discharge is a solid and sticks to the electrodes. So, it is in a position to gain or receive the electrons during electrolysis. Such type of cell is generally used for electrical vehicles, automobiles, railways, hospitals, power stations, in telephone exchange, UPS system etc.
An acid–base titration is a method of quantitative analysis for determining the concentration of an acid or base by exactly neutralizing it with a standard solution of base or acid having known concentration. A pH indicator is used to monitor the progress of the acid–base reaction.



Titration of an acid solution of unknown concentration with a base solution of known concentration



Voltage of lead-acid electrochemical cell vs. electrolyte concentration, as predicted by Nernst equation



From the above figure, it is noticed that, as the concentration of the acid decreases, voltage of the battery drops gradually. Hence maintaining the acid concentration is very important in lead acid batteries. Hence, the present experiment gains its importance in proper maintenance of the battery.

**PROCEDURE:**

**STEP-1: Preparation of standard oxalic acid solution**

0.025M Oxalic acid primary standard solution is prepared by dissolving 0.315g of Oxalic acid solid in 100 ml distilled water.

**STEP-2: Standardization of sodium hydroxide solution**

Take 10 ml of oxalic acid solution into a conical flask, add 20 ml of distilled water and a drop of phenolphthalein indicator then titrate against sodium hydroxide solution taken in a burette until color of the solution changes from colorless to pale pink. Repeat the titration for concurrent readings.

**STEP-3: Determination of acid strength of lead acid battery:**

The given test sample solution is made up to the mark using distilled water. 10 ml of this solution is transferred into a conical flask. Add 20 ml of distilled water and a drop of phenolphthalein indicator and titrated against sodium hydroxide solution taken into a burette until color of the solution changes from colorless to pale pink. Repeat the titration for concurrent readings.

**Report:** Strength of an acid in Pb-Acid battery = M

## DEMONSTRATION EXPERIMENT - A4

# DETERMINATION OF CHLORIDE CONTENT IN THE GIVEN WATER SAMPLE BY ARGENTOMETRIC METHOD

## Observation and Calculations

**Chloride Ion Concentration (mg/L) = $(A \times N \times 35.45) * 1000 / V_{sample}$**

*Where: A = volume of titrant used, N is normality of silver nitrate (here we used N/71 or 0.0141 N), and* $V_{sample}$ *is volume of sample used (mL).*

## DEMONSTRATION EXPERIMENT - A4

# DETERMINATION OF CHLORIDE CONTENT IN THE GIVEN WATER SAMPLE BY ARGENTOMETRIC METHOD

## Theory:

Chloride in the form of chloride ($Cl^-$) ion is one of the major inorganic anions in water and wastewater. The chloride concentration is higher in wastewater than in raw water because sodium chloride is a common article of diet and passes unchanged through the digestive system (Average estimate of excretion: 6 g of chlorides/person/day; additional chloride burden due to human consumption on wastewater: 15 mg/L). Along the sea coast chloride may be present in high concentration because of leakage of salt water into the sewage system. It also may be increased by industrial process. In potable water, the salty taste produced by chloride concentration is variable and depends on the chemical composition of water. Some waterscontaining 250 mg/L $Cl^-$ may have a detectable salty taste if sodium cation is present. On the other hand, the typical salty taste may be absent in waters containing as much as 1000 mg/Lwhen the predominant cations are calcium and magnesium. In addition, a high chloride contents may harm metallic pipes and structures as well as growing plants.

The measured chloride ions can be used to know salinity of different water sources. For brackish water (or sea water or industrial brine solution), it is an important parameter and indicates the extent of desalting of apparatus required. It also interferes with COD determination and thus it requires a correction to be made on the basis of amount present or else a complexing agent, such as $HgSO_4$ can be added. Further, chloride ions are used as tracer ions in column studies to model fate of different contaminants in soil and liquid media.

## Procedure

The silver nitrate solution is standardized against standard chloride solution, prepared from sodium chloride (NaCl). During the titration, chloride ion is precipitated as white silver chloride

$$\mathbf{Ag^+ + Cl^- <=> AgCl \qquad (Solubility\ product\ constant,\ K_{sp}=3\times10^{-10})}$$

The indicator (potassium chromate) is added to visualize the endpoint, demonstrating presence of excess silver ions. In the presence of excess silver ions, solubility product of silver chromate exceeded and it forms a reddish-brown precipitate (Eq.2). This stage is taken as evidence that all chloride ions have been consumed and only excess silver ions have reacted with chromate ions:

$$\mathbf{2Ag^+ + CrO_4^{2-} <=> Ag_2CrO_4 \qquad (K_{sp}=5\times10^{-12})\ (1)}$$

**Apparatus:** Burette, conical flask, pipette, measuring cylinder

**Reagents:** Potassium chromate indicator solution, standard silver nitrate titrant.

**Steps:**

1. Take 25 ml sample in a conical flask. Measure sample pH.
2. Add 1.0ml indicator solution,
3. Titrate with standard silver nitrate solution to pinkish yellow end point and note downvolume of titrant used. Also measure sample pH.
4. Calculate chloride ion concentration using:

**Precautions:**

1. A uniform sample size must be used, preferably 100 mL, so that ionic concentrationsneeded to indicate the end point will be constant.
2. The pH must be in the range of 7 to 8 as silver ions are precipitated as AgOH at high pHlevels and the chromate ions are converted to $Cr_2O_7^{2-}$ at low pH values.
3. A definite amount of indicator must be used to provide a certain concentration of chromate ions, otherwise silver chromate may form too soon or not soon enough.
4. Caution should be made to notice indicator color change as it can varies person-to-person. The usual range is 0.2 to 0.4 mL of titrant.

**Report:** Chloride content in the given water sample =

## DEMONSTRATION EXPERIMENT - A5

# DETERMINATION OF ACID VALUE OF BIOFUEL

### Determination of the Acid Number of Vegetable Oil / Biodiesel by Titration Method

## Observation and Calculations

**For example, for analysis of crude oil:**

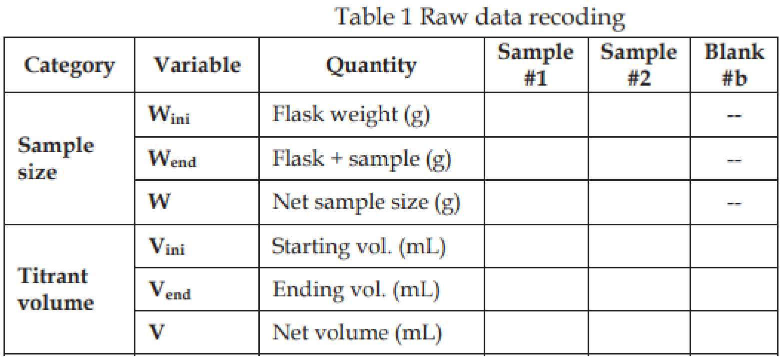
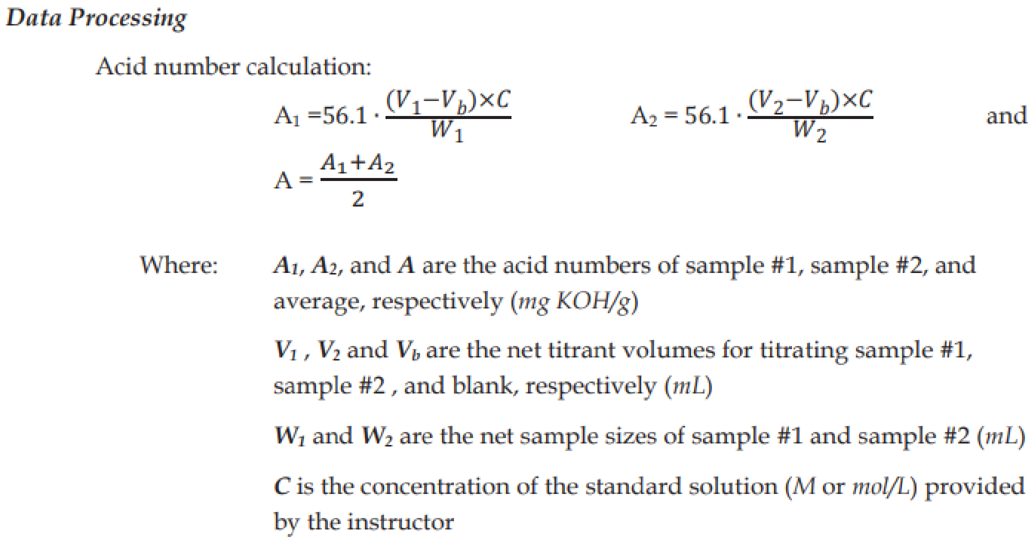


Table 1 Raw data recoding

| Category | Variable | Quantity | Sample #1 | Sample #2 | Blank #b |
|---|---|---|---|---|---|
| **Sample size** | **$W_{ini}$** | Flask weight (g) | | | -- |
| | **$W_{end}$** | Flask + sample (g) | | | -- |
| | **W** | Net sample size (g) | | | -- |
| **Titrant volume** | **$V_{ini}$** | Starting vol. (mL) | | | |
| | **$V_{end}$** | Ending vol. (mL) | | | |
| | **V** | Net volume (mL) | | | |



***Data Processing***

Acid number calculation:

$$A_1 = 56.1 \cdot \frac{(V_1 - V_b) \times C}{W_1} \qquad A_2 = 56.1 \cdot \frac{(V_2 - V_b) \times C}{W_2} \qquad \text{and}$$

$$A = \frac{A_1 + A_2}{2}$$

Where: $A_1$, $A_2$, and $A$ are the acid numbers of sample #1, sample #2, and average, respectively (*mg KOH/g*)

$V_1$ , $V_2$ and $V_b$ are the net titrant volumes for titrating sample #1, sample #2 , and blank, respectively (*mL*)

$W_1$ and $W_2$ are the net sample sizes of sample #1 and sample #2 (*mL*)

*C* is the concentration of the standard solution (*M* or *mol/L*) provided by the instructor

Similar Procedure can be applied to determine the acid number of the derived biodiesel

**Acid number of a sample:**

## DEMONSTRATION EXPERIMENT - A5

# DETERMINATION OF ACID VALUE OF BIOFUEL

### Determination of the Acid Number of Vegetable Oil / Biodiesel by Titration Method

**Theory:**

In chemistry, acid value (AV, acid number, neutralization number or acidity) is a number used to quantify the acidity of a given chemical substance. It is the quantity of base (usually potassium hydroxide (KOH)), expressed as milligrams of KOH required to neutralize the acidic constituents in 1 gram of a sample.

To make biodiesel, triglycerides (fats and oils) of oil react with an alcohol (such as methanol) to produce methyl fatty acid esters and glycerol. A catalyst such as potassium hydroxide or sodium hydroxide is added to speed up the process. However, if the oil or fat is degraded and has a lot of free fatty acids (fatty acids not attached to a glycerol backbone), these free fatty acids can react with the catalyst first to create soap, which must be removed. Therefore, before making biodiesel it is necessary to determine the amount of free fatty acids in the oil or fat, in order to know how much catalyst to add. Fats and oils with high free fatty acid levels include waste vegetable oil and animal fats. These feedstocks are cheaper to use than food-grade vegetable oil, and so are often desirable as feedstocks for making biodiesel.

A liquid fat sample combined with neutralized 95% ethanol is titrated with standardized sodium hydroxide of 0.1 eq/L normality to a phenolphthalein endpoint. The volume and normality of the sodium hydroxide are used, along with the weight of the sample, to calculate the free fatty acid value/

**Procedure:**

1. Weigh flask and record the weight on Table 1.
2. Weigh approximately 1 g of oil sample and transfer to the titration flask.
3. Record the sample size in Table 1.
4. Add 10 ml of ethanol (titration solvent) to the oil and mix the solution.
5. Add a couple of drops of the phenolphthalein indicator into the solution.
6. Swirl the solution vigorously for approximately 1 minute to ensure the uniformity of the solution.
7. Prepare a second sample for titration by repeating the steps 1 to 5.
8. Prepare a blank/ reference with only titration solvent and phenolphthalein indicator (no oil) by repeating steps
9. Fill the burette with the titrant solution (0.1 N KOH).
10. Record the starting volume of titrant on Table 1.
11. Carefully introduce the titrant from the burette into the flask while swirling the solution vigorously but without spattering.
12. Stop introducing titrant if the solution changes color to pink.
13. Record the ending volume of titrant on Table 1.
14. Repeat the steps for the second sample.
15. Repeat the steps for the blank (please note that the blank requires a much smaller volume of titrant because of its low acidity).

Similar Procedure can be applied to determine the acid number of the derived biodiesel.

**Acid number of a sample: --------------- mg KOH/** g fat /oil / biodiesel **g**

## EXERCISE EXPERIMENT – B1
## CONDUCTOMETRIC ESTIMATION OF AN ACID MIXTURE

**OBSERVATION AND CALCULATION:**

| Volume of NaOH ($cm^3$) | Conductance (mS) | Volume of NaOH ($cm^3$) | Conductance (mS) | Volume of NaOH ($cm^3$) | Conductance (mS) |
|---|---|---|---|---|---|
| 0.0 | | 3.5 | | 7.0 | |
| 0.5 | | 4.0 | | 7.5 | |
| 1.0 | | 4.5 | | 8.0 | |
| 1.5 | | 5.0 | | 8.5 | |
| 2.0 | | 5.5 | | 9.0 | |
| 2.5 | | 6.0 | | 9.5 | |
| 3.0 | | 6.5 | | 10.0 | |

Normality of NaOH = __________ N (given)

**Estimation of HCl**

Volume of NaOH at first equivalence point__________ $cm^3$ (HCl)

Volume of acid mixture = 50 $cm^3$

Therefore, $N_{HCl} = \dfrac{N_{NaOH} \; X \; V_{NaOH}}{V_{HCl}} = \dfrac{\qquad X \qquad}{50} = \qquad N$

Normality of HCl solution = N

Amount of HCl present in 1000 $cm^3$ of the solution = $N_{HCl}$ x Gram equivalent weight of HCl

= __________ X 36.5 = __________ g

Amount of HCl present in the given solution = __________ g

# EXERCISE EXPERIMENT – B1

# CONDUCTOMETRIC ESTIMATION OF AN ACID MIXTURE

**PRINCIPLE:**

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

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

## ESTIMATION OF HCL AND $CH_3COOH$

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

Now add standard NaOH solution from a burette in increments of 0.5 $cm^3$. After each addition, stir the solution gently and note down the conductance. As the titration proceeds, the conductance first gradually decreases and then rises slowly and finally rises sharply. Continue titration until the conductance more or less the same as it was in the beginning. **Draw a graph of conductance against volume of NaOH added**. The point of intersection of the first and the second lines give the volume of sodium hydroxide needed to neutralize only hydrochloric acid. The point of intersection of the second and the third straight lines give the volume of NaOH required to neutralization both HCl and $CH_3COOH$ (after drawing a perpendicular to X-axis).

**Estimation of $CH_3COOH$**

Volume of NaOH required to neutralized $CH_3COOH$ ($V_2$-$V_1$) = cm$^3$

Volume of acid mixture = 50 cm$^3$

Therefore, $N_{CH3COOH}$ = $\frac{N_{NaOH} \ X \ V_{NaOH}}{V_{CH_3COOH}} = \frac{X}{50} = \quad N$

Normality of $CH_3COOH$ solution = N

Amount of $CH_3COOH$ present in1000 cm$^3$ of the solution = $N_{CH3COOH}$ x Gram equivalent weight of

$CH_3COOH$

= __________ X 60

= __________ g

Amount of HCl and $CH_3COOH$ present in a given acid mixture = __________g and __________g

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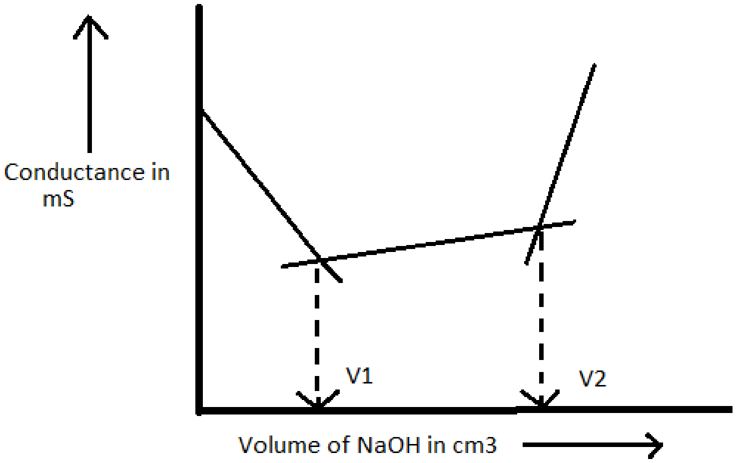


**Report :** Amount of HCl and $CH_3COOH$ present in a given acid mixture =-----------g and ---------g

## EXERCISE EXPERIMENT – B2

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

### OBSERVATION AND CALCULATION:

| Volume of $K_2Cr_2O_7$ in cm$^3$ | $\Delta V$ | E in mV | $\Delta E$ | $\Delta E/\Delta V$ | Volume of $K_2Cr_2O_7$ in cm$^3$ | $\Delta V$ | E in mV | $\Delta E$ | $\Delta E/\Delta V$ |
|---|---|---|---|---|---|---|---|---|---|
| 0.0 | 0.5 cm$^3$ | | === | ==== | 5.5 | 0.5 cm$^3$ | | | |
| 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 | | | | | | | | | |

**Calculations:**

$V_1.N_1 = V_2.N_2$ Where $V_1$ = Volume of $K_2Cr_2O_7$ at the equivalence point (from the graph)

$N_1$ = Normality of $K_2Cr_2O_7$ solution = __________N (given)

$V_2$ = Volume of FAS solution = 25 cm$^3$

$N_2$ = Normality of FAS solution = __________ N

$$\therefore N_2 = \frac{V_1 X N_1}{V_2} = \frac{X}{25} = ----------N$$

Mass per dm$^3$ = Normality x Gram equivalent mass

Mass of FAS per dm$^3$ = Normality of FAS x Gram equivalent mass of FAS

= ______ x 392 g

= ________ g

**REPORT:** Mass of FAS present in one dm$^3$ of solution = __________ g.

**Date:** **Signature of the faculty**

## EXERCISE EXPERIMENT – B2

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

**PRINCIPLE:** Redox titrations can be carried out potentiometrically using platinum-calomel electrode combination. For the reaction: Reduced form → Oxidized form + ne$^-$,

$$E = E^o + \frac{0.0591}{n}\log\frac{[oxidized\ form]}{[\mathrm{Re}\,duced\ form]}$$

The potential, E, is given by Nernst equation,

Where, $E^o$ is the standard potential of the system, and [X] represent the molar concentration x.
Suppose that, in beaker we have acidified $Fe^{2+}$ solution, and we add slowly $K_2Cr_2O_7$ from a burette, then following reaction takes place.

$$6\,Fe^{2+} + Cr_2O_7^{2-} \rightarrow 6\,Fe^{3+} + 2Cr^{3+}$$

Before the equivalence point, the potential is determined by the $Fe^{2+}$/ $Fe^{3+}$ system.

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

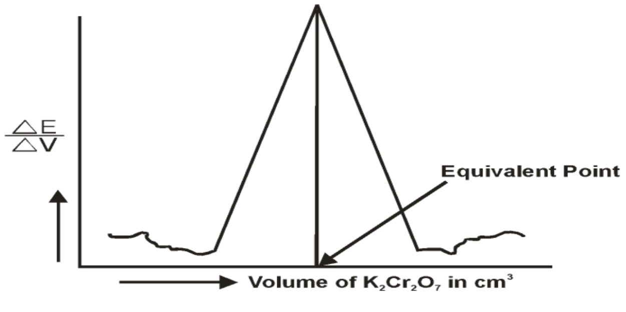
The potential of the solution will be around 0.75V (since the contribution from the second term is negligible).
After the equivalence point, the potential is determined by the $Cr_2O_7^{2-}/Cr^{3+}$ system.

$$E = E^o + \frac{0.0591}{6}\log\frac{[Cr_2O_7^{\ 2-}]}{[Cr^{3+}]} = 1.33V + 0.00985\log\frac{[Cr_2O_7^{\ 2-}]}{[Cr_{3+}]}$$

The potential of the solution will be around 1.3V
At the equivalence point, the potential is average potential of both systems.
Thus, there is an abrupt increase in potential of the solution near the end point**.**

**PROCEDURE:** Add 25 cm$^3$ of ferrous ammonium sulfate (FAS) solution into a 100 ml beaker. Add 25 cm$^3$ of dilute Sulphuric acid. Immerse the platinum - calomel electrodes assembly in the solution. Measure the potential by adding $K_2Cr_2O_7$ solution from the burette in increments of 0.5 cm$^3$. stir the solution gently and note down the emf. Measure the potential of each addition.

Plot a graph of ΔE/ ΔV against volume of $K_2Cr_2O_7$ as shown in the figure. Find the equivalence point from the graph. Knowing the equivalence point, calculate the normality of the FAS solution and determine the amount of FAS per liter of the solution



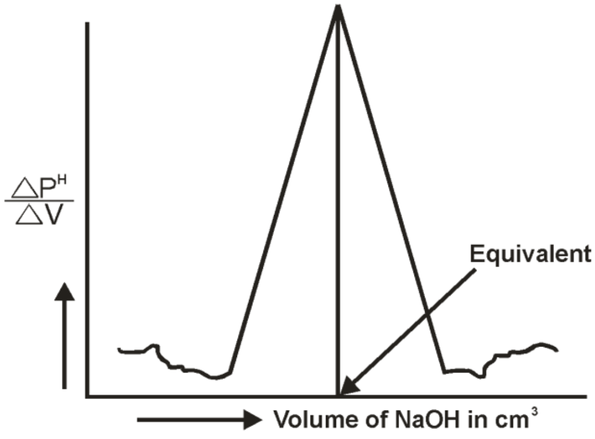
**REPORT:** Mass of FAS present in one dm$^3$ of solution =---------------g

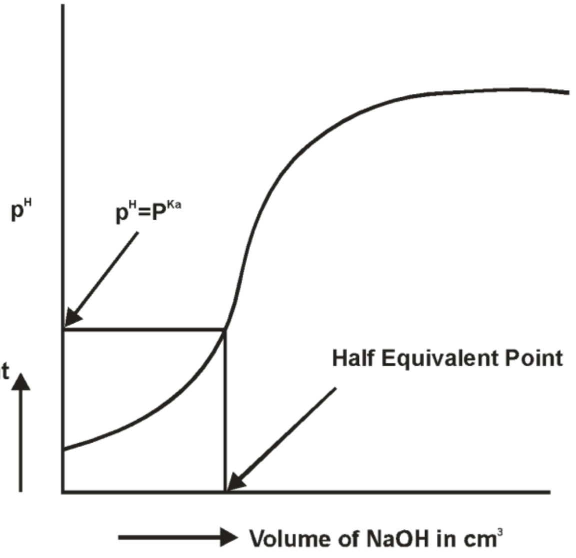
## EXERCISE EXPERIMENT – B3

## DETERMINATION OF $P^{Ka}$ OF VINEGAR USING $P^H$ SENSOR ( GLASS ELECTRODE)

**OBSERVATION AND CALCULATION:**

| Volume of NaOH in cm$^3$ | $\Delta V$ | $P^H$ | $\Delta P^H$ | $\Delta P^H/\Delta V$ | Volume of NaOH, in cm$^3$ | $\Delta V$ | $P^H$ | $\Delta P^H$ | $\Delta P^H/\Delta V$ |
|---|---|---|---|---|---|---|---|---|---|
| 0.0 | 0.5 cm$^3$ |  | ==== | ====== | 5.5 | 0.5 cm$^3$ |  |  |  |
| 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 |  |  |  |  |  |  |  |  |  |





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## EXERCISE EXPERIMENT – B3

## DETERMINATION OF $P^{Ka}$ OF VINEGAR USING $P^{H}$ SENSOR ( GLASS ELECTRODE)

**PRINCIPLE:**

Vinegar is an aqueous solution of acetic acid and trace compounds that may include flavorings. Vinegar typically contains 5–8% acetic acid by volume. Usually, the acetic acid is produced by a double fermentation, converting simple sugars to ethanol using yeast, and ethanol to acetic acid by acetic acid bacteria.[2] Many types of vinegar are available, depending on source materials.

A weak acid is an acid, which dissociated partial in solution. Example acetic acid $CH_3COOH$. When we make a solution of this acid, a part of the acid molecules dissociate.

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

For this reaction, the equilibrium constant, Ka, is given by the equation:

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

'Ka' is also known as acid dissociation constant.

The negative logarithm to base 10 of Ka is called $P^{Ka}$. ie., $P^{Ka} = -\log_{10} Ka$.

Consider a solution of a vinegar ; say acetic acid, in a beaker. Let 'Ka' be the acid dissociation constant. Let us partially neutralize the acid by adding a base, say, NaOH from a burette. Addition of base to the acid result in the formation of salt and water. The pH of the partial neutralized solution is related to $P^{Ka}$ of the acid by the Henderson-Hasselbalcs equation,

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

If we titrate the acid against NaOH, the pH of the mixture in the beaker continuously changes. When we plot a graph of pH vs. volume of NaOH, we get a 'S' shaped curve. We find that there will be sharp jump in pH at the equivalence point. At half equivalence point, [Salt] = [Acid]. Thus, according to the Henderson equation pH becomes equal to pKa at half equivalence point.

**PROCEDURE:** Add 25 $cm^3$ of the given vinegar sample into a 100 $cm^3$ beaker and add one test tube of water. Immerse the combined glass electrode into the acid. Connect the electrode terminals to a pH meter. Measure the pH of the acid. Add NaOH solution from a micro burette in increments of 0.5 $cm^3$. After each addition, stir the solution and measure the $P^H$. (After the jump in the pH, take six more readings).

Plot a graph of $\Delta P^H/\Delta V$ against volume of NaOH and determine the equivalence point. Plot another graph pH/ volume of NaOH, and note the pH at half equivalence point (Which is nothing but $P^{Ka}$).

**REPORT**: The $P^{Ka}$ of the given vinegar sample =

## EXERCISE EXPERIMENT – B4

## ESTIMATION OF TOTAL HARDNESS OF WATER BY EDTA METHOD

### OBSERVATION AND CALCULATION

### Part-A: Preparation of EDTA solution

| | |
|---|---|
| Weight of the weighing bottle +EDTA = | g |
| Weight of the weighing bottle = | g |
| Weight of the EDTA salt transferred = | g |

.

$$Molarity\ of\ EDTA\ solution = \frac{Weight\ of\ EDTA\ X\ 4}{Gram\ molecular\ wt.of\ EDTA} = \frac{\qquad\qquad X\ \ 4}{372.24}$$

= _______________ M (*a*)

**PART-B: Estimation of hardness.**

**Burette :** EDTA solution

**Conical flask:** 25cm$^3$ of hard water + $NH_3$-$NH_4Cl$ buffer

**Indicator :** 2-3 drops of Eriochrome black –T

**End point :** Wine red to pure blue

| **Burette readings** | **Trial I** | **Trial II** | **Trial III** |
|---|---|---|---|
| Final | | | |
| Initial | | | |
| Volume of EDTA run down (in cm$^3$) | | | |

Volume of EDTA used: __________ cm$^3$ (b)

1000cm$^3$of 1M EDTA = 100 g of $CaCO_3$

Therefore *b* cm$^3$of *a* molar EDTA = $\frac{b\ X\ a\ X\ 100}{1000}$ = ________________ (c) g of $CaCO_3$

25 cm$^3$ of hard water contains = __________ (c) g of $CaCO_3$

Therefore 10$^6$cm$^3$ 0f hard water contains = $\frac{cx10^6}{25} = -------ppm$

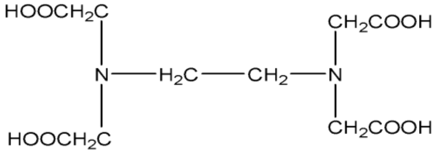
Total hardness of Water = __________ ppm of $CaCO_3$

**Date:** **Signature of the Faculty**

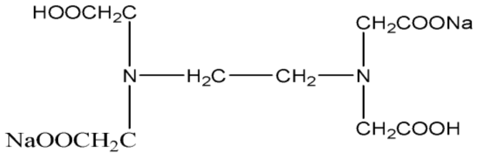
## EXERCISE EXPERIMENT – B4

## ESTIMATION OF TOTAL HARDNESS OF WATER BY EDTA METHOD

**THEORY**: Hardness of water is mainly due to the presence of calcium and magnesium salts in it. Total hardness is the sum of temporary hardness (due to bicarbonates of calcium and Magnesium) and permanent hardness (due to chlorides, sulphates etc., of Calcium and Magnesium). Ethylene diamine tetra acetic acid (EDTA) is a reagent, which reacts with metal ions like $Ca^{2+}$&$Mg^{2+}$ forming complex compounds. Therefore this reagent can be used to determine the concentration of hardness causing substances.



EDTA



$Na_2$EDTA salt

The completion of the reaction (end point of the titration) is identified using Eriochrome black- T indicator. This is an organic dye, blue in colour. It also forms relatively less stable complexes with bivalent metal ion of Ca & Mg etc., which are wine red in colour. Therefore, addition of the indicator to hard water produces wine-red Colour. When EDTA is added to hard water, it first reacts with free metal ions and then attacks the metal-indicator complex. The latter reaction can be represented as
$M^{2+}$Indicator complex + EDTA→ $M^{2+}$ EDTA c omplex (COLOURLESS) + free
Indicator (Blue) so at the end point a change from wine red to blue color is observed. Since the reaction involves the liberation of $H^+$ ions and the indicator is sensitive to the concentration of $H^+$ ions (pH) of the solution a constant pH of around 10 has to be maintained. For this purpose ammonia-ammonium chloride buffer is used.

### PROCEDURE

### Part-A: Preparation of standard EDTA solution

Weigh the weighing bottle containing disodium salt of EDTA accurately and transfer the salt in to the funnel placed on a 250 $cm^3$ volumetric flask. Weigh the bottle again .The difference between the two weights will give the amount of EDTA transferred. Pour small quantities of water over the salt on the funnel and transfer the salt in to the flask. Wash the funnel with the same water 3-4 times; Dissolve the salt by adding 5ml 1:1 Ammonia and make up the solution to the mark and shake well for uniform concentration

### Part-B: Estimation of hardness of water

Pipette out 25 $cm^3$ of the given sample of hard water in to a clean conical flask .Add 5 ml of $NH_3$-$NH_4Cl$ buffer followed by 3-4 drops of Eriochrome black T indicator. Titrate this against EDTA taken in a burette till the colour changes from wine red to pure blue .Note down the burette reading and repeat the titration to get concordant values.

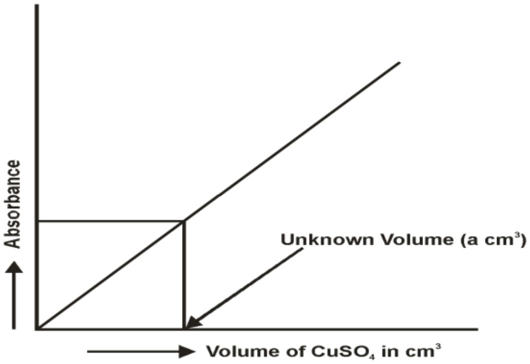
**REPORT:** Total hardness of water = _________ ppm of $CaCO_3$

## STRUCTURED EQUIRY EXPERIMENT – C1

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

### OBSERVATION AND CALCULATIONS:

| Sl.No of volumetric flask | Volume of $CuSO_4$ in $cm^3$ | Volume of ammonia solution in $cm^3$ | Concentration of copper =1.018 mg x vol. of $CuSO_4$ solution | Absorbance |
|---|---|---|---|---|
| 1 | 0.0 (Blank sol.) | 2.0 | | |
| 2 | 2.0 | 2.0 | | |
| 3 | 4.0 | 2.0 | | |
| 4 | 6.0 | 2.0 | | |
| 5 | 8.0 | 2.0 | | |
| 6 | Electroplating effluent sample | 2.0 | | |



1000 $cm^3$ of stock solution contains 4 g of $CuSO_4 . 5H_2O$
249.54 g of $CuSO_4.5H_2O$ ≡ 63.54 g of Cu
4 g of $CuSO_4.5H_2O$ ≡ 63.54 x 4 / 249.54 = 1.018 g of Cu per 1000 $cm^3$ of stock solution

∴ 1 $cm^3$ of $CuSO_4.5H_2O$ ≡ 1.018/1000 = 0.001018 g of Cu = 1.018 mg of Cu

Volume of $CuSO_4$ in the unknown solution = _________$cm^3$

∴ Cu present in 'a' $cm^3$ of electroplating effluent sample = 'a' $cm^3$ x 1.018 mg = mg

**Date:** **Signature of the faculty**

## STRUCTURED EQUIRY EXPERIMENT – C1

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

**Theory:**

E-waste", "electronic waste", "e-scrap" Are used electronics that are nearing the end of their useful life, and are discarded. Waste printed circuit boards (PCBs). PCBs comprise metals (Cu, Sn, Pb, Ag, Au, Pd, Fe, Ni, and Cr), nonmetals (glass fibers, electronic component insulators, capacitors, resistors, and so on), and organic compounds (epoxy resin, paints...). Copper is the primary component of PCB, which is employed as an electric current conductor. These discarded metals without treatment threaten the economy, the environment, and human health.

**PRINCIPLE:** When a monochromatic light of intensity Io is incident on a colored solution, a part ($I_a$) of it is absorbed, a part ($I_r$) is reflected and the remaining part ($I_t$) is transmitted.
Thus, $I_o = I_a + I_r + I_t$

Absorbance is given as $A = \log \frac{I_o}{I_t}$
According to Beer- Lambert's law, $A = \epsilon c l$
Where, $\epsilon$ = molar extinction coefficient, a constant for any particular colored substance for a given wave length of light,
c = Molar concentration of the solution and
l = path length.

When the path length is kept constant, then $A \alpha c$. Hence a plot of absorbance, A, against concentration c, gives a straight line.
Chemical analysis through measurements of absorption of light of a particular wavelength is known as colorimetry. The absorbance of light of a particular wavelength by a substance in solution varies directly with its concentration and the thickness of the solution. When the thickness of the medium is kept constant, the absorbance directly depends upon the concentration.

A series of solutions with different concentrations of cuprammonium ions is prepared and absorbance of each is measured at **620 nm** radiation. A calibration graph is obtained. The absorbance of cuprammonium ions of unknown solution is also measured and the unknown volume is determined using the calibration graph.

**PROCEDURE:**

All the detachable components free from copper (e.g., relays, capacitors, and resistance) were separated, the bare PCB boards were shredded and crushed by a cutting and grinding machine and passed through a sieve mesh to obtain the WPCB powder of varying particle sizes. Then the mixer of WPCB powder and $K_2S_2O_8$ ( with suitable mass ratios) together with grinding balls, were sealed in the $ZrO_2$ pot. The mixture was then co-ground at different rotary speeds under ambient conditions for different periods of time (Stage I). Co-ground products of PCB powder and $K_2S_2O_8$ is leached in

deionized water with constant magnetic stirring at different temperatures: 25, 35, 45, and 55 °C. The leaching solution and solid residues were rapidly separated by vacuum filtration after the reaction was

complete. (Stage-II). After filtration, $CuSO_4 \cdot 5H_2O$ could be easily separated from $K_2SO_4$ solution because of their differences in solubility. (Stage-III).
Take six 50 $cm^3$ volumetric flasks. Transfer 0, 2, 4, 6 and 8 $cm^3$ of $CuSO_4$ to first five flasks. Take the above extracted electroplating effluent unknown solution in the six flasks. Add 2 $cm^3$ of ammonia solution to each one of the six flasks. Dilute up to the mark and mix well. After 10 minutes, set the absorbance of first solution to zero at **620 nm** radiations in the instrument. Then, measure the absorbance of remaining five solutions with the same settings.

Draw a calibration curve by volume of $CuSO_4$ on x-axis and absorbance on y- axis. (Draw a straight line passing through the origin). Using the graph and knowing the absorbance of six solutions, find out the volume of $CuSO_4$ in the sixth flask.

**REPORT**: Mass of Cu in the Electroplating effluent sample = mg

## STRUCTURED EQUIRY EXPERIMENT – C2

# DETERMINATION OF VISCOSITY CO-EFFICIENT OF LUBRICANT USING OSTWALD’S VISCOMETER

## OBSERVATION AND CALCULATION:

| Liquid | Time of Flow (seconds) | Average Time of Flow (seconds) |
|---|---|---|
| Given Lubricant Liquid (l) | | **$t_l$** = |
| | | |
| | | |
| Water (w) | | **$t_w$** = |
| | | |
| | | |

Lab temperature = __________ °C

$d_l$ (density of organic liquid) = __________ g $cm^{-3}$

$d_w$ (density of water) = __________ g $cm^{-3}$

$\eta_w$ (Viscosity coefficient of water) = __________ milli poise

$$\frac{\eta_1}{\eta_w} = \frac{t_1 d_1}{t_w d_w}$$

$$\eta_1 = \frac{t_1 d_1}{t_w d_w} X \eta_w = \frac{\qquad X \qquad X \qquad}{X} = \text{..........................} \mathit{milli\ poise}$$

**REPORT**: Viscosity coefficient of the given Lubricant liquid = ……………… milli poise.

**Date:** **Signature of the Faculty**

## STRUCTURED EQUIRY EXPERIMENT – C2

## DETERMINATION OF VISCOSITY CO-EFFICIENT OF LUBRICANT USING OSTWALD'S VISCOMETER

**PRINCIPLE:** Viscosity arises due to friction between moving layers of a liquid. A liquid flowing through a cylindrical tube of uniform diameter is expected to move in the form of molecular layers. Layer close to the surface is almost stationary while that t the axis of the tube moves faster than any other intermediate layer. A slow moving layer excerts a drag or friction on its nearest moving layer backwards. This property of the liquid, which retards or opposes the motion between the layers, is called viscosity. The Coefficient of viscosity is defined as the tangential force per unit area required maintaining a unit velocity gradient between the two successive layers of the liquid situated unit distance apart. The Coefficient of viscosity of a liquid is given by the Poiseuille's formula.

$$\eta = \frac{\pi pr^4 t}{8Vl}$$

Where 'v' is the volume of the liquid, 'r' is the radius of the tube and 'p' is the pressure between the two ends of the tube is the Coefficient of viscosity. If equal volumes of the two different liquids are allowed to flow through the same tube under identical conditions then,

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

The time't' taken by the given liquid to travel through a certain distance in the tube is determined. The time't' taken by standard liquid to travel through the same distance is measured. Knowing the densities of the two liquids ($d_1$ and $d_2$) and also the coefficient of viscosity of the standard liquid, coefficient of viscosity of test liquid is calculated.

**PROCEDURE:** Take a dry viscometer. (Do not wash!) Attach a rubber to the narrow limb. Immerse the viscometer in water bath and fix it vertically to a stand. Using a pipette, transfer 10 $cm^3$ of the given liquid into the wider limb of the viscometer. Suck the lubricant liquid and fill the bulb on the narrow limb slightly above the upper mark. Allow the liquid to flow down through the capillary. Start a stop clock when the level of the liquid crosses the lower mark. Note down the time of flow.

Remove the viscometer from the stand. Remove the rubber tube. Pour out the liquid from the viscometer into the beaker. Using acetone (through a dropper) rinse the viscometer. Dry it in air over for 20 minutes. Take out the viscometer and follow a similar procedure for determining the average time of flow for deionized water. (Use a different pipette for water).

Using a thermometer note the temperature of the water bath. This is lab temperature.

From your teacher, get the values of $d_1$ (density of organic liquid), $d_2$(density of water) and $\eta_2$ (Viscosity coefficient of water)

Find $\eta_1$ (viscosity coefficient of organic liquid) using the relation.

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

**REPORT**: Viscosity coefficient of the given lubricant liquid = ------------ milli poise.

## STRUCTURED EQUIRY EXPERIMENT – C3

### OBSERVATION AND CALCULATION:

### Part A: Preparation of potassium dichromate solution

| | |
|---|---|
| Weight of the weighing bottle + $K_2Cr_2O_7$ = | g |
| Weight of the weighing bottle = | g |
| Weight of the $K_2Cr_2O_7$ salt transferred = | g |

$$Normality\ of K_2Cr_2O_7\ solution = \frac{Wt.of\ K_2Cr_2O_7\ X\ 4}{Gram\ Eq.wt.of\ K_2Cr_2O_7} = \frac{\qquad X\ 4}{49.06}$$

= N (***a***)

### Part B: estimation of Iron:

**Burette :** $K_2Cr_2O_7$ solution

**Conical flask:** 25 $cm^3$ of TMT bar solution +5 $cm^3$ of conc. HCl, heat nearly to Boiling add stannous chloride from a dropper until colorless solution is Obtained. Cool the solution to room temperature. Add 2tt of DM water. Add 5 $cm^3$ of mercuric chloride at a stretch.

**Indicator :** Drops of potassium ferricynide on wax paper (External indicator)

**End point :** No blue coloration when a drop of the mixture is mixed with a drop of indicator

| Burette readings | Trail I | Trail II | Trail III |
|---|---|---|---|
| Final | | | |
| Initial | | | |
| Volume of $K_2Cr_2O_7$ run down (in $cm^3$) | | | |

**CALCULATION**

**Volume of $K_2Cr_2O_7$ used = .....................................$cm^3$ (b)**

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

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

$$\text{'b' } cm^3 \text{ of 'a' N } K_2Cr_2O_7 \text{ solution} = \frac{55.85\ X\ a\ X\ b}{1000\ x\ 1}$$

**= -------------g (C) of Fe/ 25 $cm^3$ of solution**

**Percentage of iron in the TMT bar sample =** $\frac{C\ X\ 100}{Weight\ of\ TMT\ bar}$

## STRUCTURED EQUIRY EXPERIMENT – C3

## ESTIMATION OF PERCENTAGE OF IRON IN TMT BAR BY EXTERNAL INDICATOR METHOD

### Principle:

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

**Part A: Preparation of standard $K_2Cr_2O_7$ solution**

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

**Part B: Estimation of Iron**

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

$$\underset{\text{Yellow}}{2FeCl_3} + SnCl_2 \rightarrow \underset{\text{Colorless}}{2FeCl_2} + SnCl_4$$

$$SnCl_2 + 2HgCl_2 \rightarrow SnCl_4 + \underset{\text{Silky whiite}}{Hg_2Cl_2}$$

$$K_2Cr_2O_7 + 8\,HCl \rightarrow 2KCl + 2\,CrCl_3 + 4H_2O + 3[O]$$

$$\underline{(2FeCl_2 + 2\,HCl + [O] \rightarrow 2\,FeCl_3 + H_2O) \times 3}$$

$$K_2Cr_2O_7 + 14\,HCl + 6FeCl_2 \rightarrow 2KCl + 6FeCl_3 + \underset{\text{Green}}{2CrCl_3} + 7\,H_2O$$

**REPORT**: Percentage of iron in TMT bar = --------------%

## STRUCTURED EQUIRY EXPERIMENT – C4

# DETERMINATION OF CHEMICAL OXYGEN DEMAND OF INDUSTRIAL WASTE WATER SAMPLE

**OBSERVATION AND CALCULATION**:

**PART A: Preparation of Ferrous ammonium sulphate (FAS) solution**:

| | |
|---|---|
| Weight of the weighing bottle + FAS = | g |
| Weight of the weighing bottle = | g |
| Weight of the FAS salt transferred = | g |

Normality of FAS solution = $\frac{Wt.of\ FAS\ X4}{Gram\,eq.wt.of\ FAS} = \frac{\quad X4}{392} = ..............N(a)$

Volume of FAS consumed in the blank titration = …………… $cm^3$ (**b**)

**Part-**B**: Back titration:**

**Burette** **:** FAS solution

**Conical flask:** 25$cm^3$ of waste water sample +25 $cm^3$ of potassium dichromate
+ 10 $cm^3$ of 1:1 Sulphuric acid containing mercuric sulphate and silver Sulphate

**Indicator** **:** 3-4 Drops of ferroin indicator.

**End point** **:** Blue green to reddish brown

| **Burette readings** | **Trial I** | **Trial II** | **Trial III** |
|---|---|---|---|
| Final | | | |
| Initial | | | |
| Volume of FAS run down (in $cm^3$) | | | |

Back titrate valve = _________ $cm^3$ (**c**)

Amount of potassium dichromate (in terms of FAS) that has reacted with water sample = (**b**)-(**c**) $cm^3$
=……..$cm^3$

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

***b* -*c*** $cm^3$ of ***a*** N FAS solution = $\frac{(b-c)XaX8}{1000} = \frac{\qquad}{1000}$

25 $cm^3$ of wastewater requires (***d***) g of oxygen= ……… g of oxygen (***d***)

Therefore, 1000 $cm^3$ of waste water requires = $\frac{dx1000}{25}$ = ……….. g oxygen

COD of the given sample of water = ___________ $mg/dm^3$ of oxygen

**Date:** **Signature of the faculty**

## STRUCTURED EQUIRY EXPERIMENT – C4

# DETERMINATION OF CHEMICAL OXYGEN DEMAND OF INDUSTRIAL WASTE WATER SAMPLE

**THEORY:** COD is a measure of oxygen equivalent of that portion of oxidisable materials that can be oxidized by a strong oxidizing agent. Chemical oxygen demand is an important parameter in industrial wastewater treatment. Straight chain aliphatic compounds, aromatic hydrocarbons, straight chain alcohol, acids, pyridine and other oxdisable material are present as impurities in wastewater. Straight chain compounds, acetic acid etc. are oxidisabe more effectively when silver sulphate is added as catalyst. Addition of mercuric sulphate would help avoid interference of chloride ions.

**PROCEDURE:**

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

Weigh accurately the given FAS and transfer it into a 250 $cm^3$ standard flask using a funnel. Add 30$cm^3$ of dilute sulphuric acid followed by about 100 $cm^3$ of water. Dissolved, make it up to the mark and shake well for uniform concentration.

**Part-B: Blank titration**:

Pipette out 25$cm^3$ of potassium dichromate into a conical flask-using pipette. Add 10 $cm^3$ of 1:1 sulphuric acid containing mercuric sulphate and silver sulphate and 3 drops ferroin indicator. Titrate against FAS taken in the burette until the colour changes from blue green to reddish brown. Note the burette reading and repeat the titration to get concordant values.

**Part-C: Back titration:**

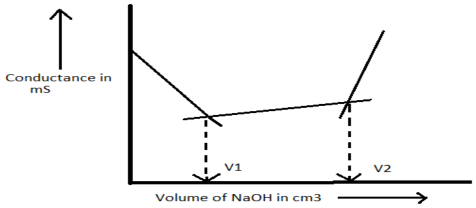
Pipette out 25 $cm^3$ of given sample of waste water into a conical flask. Add 25 $cm^3$ of standard potassium dichromate solution using a pipette. Add 10 $cm^3$ of 1:1 sulphuric acid containing mercuric sulphate and silver sulphate while shaking the flask constantly. Reflux the content of flask for 30 minutes. Cool to room temperature. Add 3-4 drops ferroin indicator and Titrate against FAS solution taken in the burette until the colour changes from bluish green to reddish brown. Note down the burette reading and repeat the titration to get concordant values.

**REPORT**: COD of the given sample of water = __________ mg/$dm^3$ of oxygen

# BRIEF PROCEDURE WRITE UP

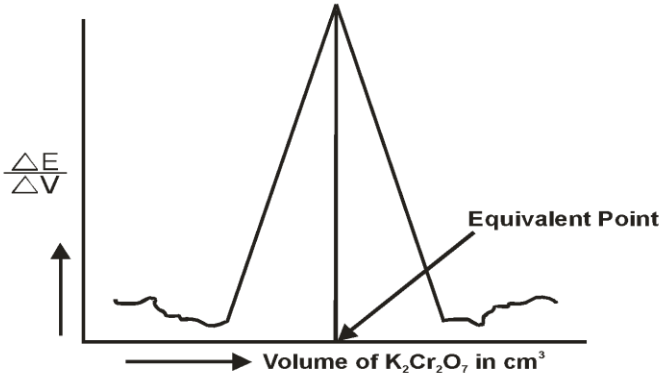
## B1: CONDUCTOMETRIC ESTIMATION OF THE ACID MIXTURE

1. Fill the micro burette with NaOH solution.
2. Pipette out 50 $cm^3$ of given acid mixture into a 100 $cm^3$ beaker. The conductivity cell is placed in it. The cell is connected to a conductivity meter and the conductance is measured. Now add NaOH in increments of 0.5 $cm^3$ and measure the conductance after each addition.
3. Plot a graph of Conductance against volume of NaOH. The point of intersection of the first and second lines give the volume of NaOH needed to neutralize only HCL. The point of intersection of the second and third lines give the volume of NaOH needed to neutralize only HCl and $CH_3COOH$. From the normality and volume of NaOH, the normality and amount of HCL and $CH_3COOH$ in the given solution are calculated.



## B2: POTENTIOMETRIC ESTIMATION OF FAS USING STANDARD $K_2Cr_2O_7$ SOLUTION

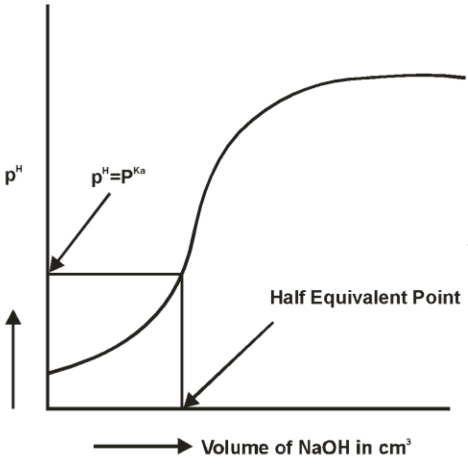
1. Fill the micro burette with $K_2Cr_2O_7$solution
2. Add 50 $cm^3$ of ferrous ammonium sulfate (FAS) solution into a 100 ml beaker. Add one test tube full of dilute Sulphuric acid. Immerse the platinum - calomel electrodes assembly in the solution. Measure the potential by adding $K_2Cr_2O_7$ solution from the burette in increments of 0.5 $cm^3$. Stir the mixture carefully. Measure the potential of each addition.
3. Plot a graph of $\Delta E/ \Delta V$ against volume of $K_2Cr_2O_7$ and determine the equivalence point.

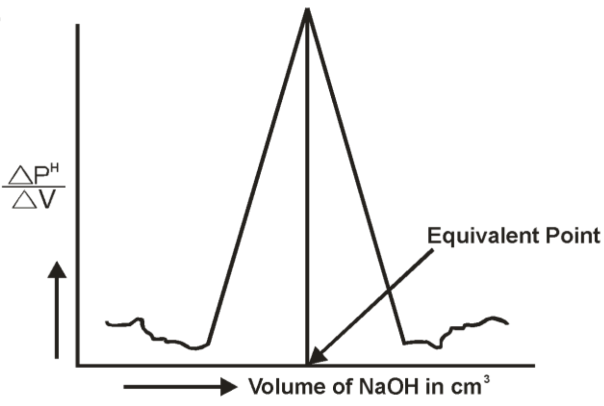


4. Calculate the normality of FAS. Calculate the mass of FAS in $dm^3$ of solution.

## B3: DETERMINATION OF $P^{Ka}$ OF VINEGAR USING $P^{H}$ SENSOR ( GLASS ELECTRODE)

1. Fill the micro burette with NaOH solution.
2. Add 25 $cm^3$ of the given vinegar into a 100 $cm^3$ beaker. Immerse the combined glass and calomel electrode into the acid. Connect the electrode terminals to a pH meter. Measure the pH of the acid. Add NaOH solution from a micro burette in increments of 0.5 $cm^3$. After each addition, stir the solution and measure the $P^H$.
3. Plot a graph of $\Delta pH/ \Delta V$ against volume of NaOH and determine the equivalence point. Plot another graph of pH against volume of NaOH and determine the pKa of the weak acid.





## B4: ESTIMATION OF TOTAL HARDNESS OF WATER BY EDTA METHOD.

**Preparation of standard disodium salt of EDTA:**

Weigh the given $Na_2EDTA$ in to a 250 $cm^3$ of volumetric flask. Add 5 $cm^3$ of 1:1ammonia. Dissolved the crystals in demineralization water. Make up to the mark and shake well.

$$Molarity\ of\ Na_2EDTA = \frac{Weight\ of\ Na_2EDTA\ X\ 4}{Gram\ molecular\ weight\ of\ Na_2EDTA}\ (372.24)$$

**Determination of total hardness of water:**

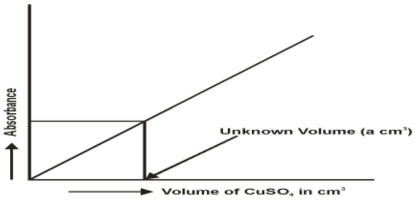
**Burette :** EDTA solution
**Conical flask:** 25$cm^3$ of hard water +5 $cm^3$ of buffer ($NH_3$-$NH_4Cl$)
**Indicator :** 2-3 drops of Eriochrome black –T
**End point :** Wine red to pure blue

Calculate the hardness of water knowing the volume of $Na_2$ EDTA consumed.

## STRUCTURED EQUIRY EXPERIMENTS

### C1: ESTIMATION OF COPPER PRESENT IN ELECTROPLATING EFFLUENT BY OPTICAL SENSOR (COLORIMETRY)

1. From a burette, take 2, 4, 6, 8 $cm^3$ of given copper sulphate solution into four separate 50 $cm^3$ volumetric flask. From another burette, add 2 $cm^3$ of ammonia to each one of them, to the extracted electroplating effluent solution and blank solution flask. To the entire six flasks, add water up to the mark. Mix well. Wait for 10 minutes. In the colorimeter set the wavelength of light to 620 nm. For the blank solution, set absorbance to zero. Then, measure the absorbance of other five solutions.
2. Plot a graph of absorbance against volume of $CuSO_4$. Determine the volume of $CuSO_4$ in unknown solution from the graph
3.



4. Calculate the mass of Cu in the extracted electroplating effluent solution

### C2: DETERMINATION OF VISCOSITY CO-EFFICIENT OF LUBRICANT USING OSTWALD'S VISCOMETER.

15 $cm^3$ of given organic liquid is pipetted out into a wider limb of the Oswald viscometer and the liquid is sucked through the other limb. The time of flow between two fixed points one above and (one below the bulb in the narrow limb of the viscometer) is measured. The measurement is repeated for three times and the average time of flow of lubricant liquid is calculated. The liquid is removed; the viscometer is washed with acetone and dried in an electric oven. 15 $cm^3$of water is pipetted into the wider limb and the average time of flow or water is determined as before. From the density of the liquid and of water, and the viscosity coefficient of water, the viscosity co-efficient of the given organic liquid is determined.

$$\eta_1 = \frac{t_l d_l}{t_w d_w} \times \eta_w$$

Where, $\eta_l$ = Viscosity coefficient of the given lubricant liquid
$t_l$ = Time of free flow of liquid
$d_l$ = Density of organic liquid
$t_w$ = Time of free flow of water
$d_w$ = Density of water
$\eta_w$ = Viscosity coefficient of water.

## C3 : ESTIMATION OF PERCENTAGE OF IRON IN TMT BAR BY EXTERNAL INDICATOR METHOD

**Preparation of standard Potassium dichromate ($K_2Cr_2O_7$):**

Weigh the given $K_2Cr_2O_7$ in to a 250cm$^3$ of volumetric flask. Dissolved the crystals in demineralized water. Make up to the mark and shake well

$$Normality\ of\ K_2Cr_2O_7\ solution = \frac{Weight\ of\ K_2Cr_2O_7 X 4}{Gram\ euivalent\ wt, of\ K_2Cr_2O_7} \ (49)$$

**Determination of Percentage of Iron in Haematite ore:**

**Burette** **:** $K_2Cr_2O_7$ solution

**Conical flask:** 25 cm$^3$ of TMT bar solution +5 cm$^3$ Conc. HCl, heat + stannous chloride drop wise till colurless, 2 drops excess. Cool + 5 cm$^3$ of mercuric chloride, shake well to get a silky white ppt + 2 test tube of water

**Indicator** **:** Potassium ferricyanide – (**External indicator**)

**End point** **:** Indicator fails to give blue colour.

Calculate the Percentage of Iron in TMT bar solution by knowing the volume of $K_2Cr_2O_7$ consumed

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## C4 : DETERMINATION OF COD OF INDUSTRIAL WASTE WATER SAMPLE

**Preparation of standard ferrous ammonium sulphate (FAS):**

Weigh the given FAS in to a 250 cm$^3$ of volumetric flask. Add 30 cm$^3$ of 4N $H_2SO_4$. Dissolved the crystals in demineralized water. Make up to the mark and shake well

$$Normality\ of\ FAS\ solution = \frac{Weight\ of\ FAS\ X\ 4}{Gram\ euivalent\ wt. of\ FAS} \ \underline{(392)}$$

**Back Titration:**

**Burette** : Standard FAS solution

**Conical flask**: 25 cm$^3$ of waste water +25 cm$^3$ of $K_2Cr_2O_7$ + 1g of $HgSO_4$ + 1g of $Ag_2SO_4$ + 10 cm$^3$ of 1:1 $H_2SO_4$. Reflux (if necessary) for 30 minutes, cool + 3 drops of indicator

**Indicator** : 3-4 drops of ferroin

**End point** **:** Blue-green to reddish brown

***Do a blank titration without wastewater as above***

Calculate the COD of waste water sample by knowing the difference in titre values

# VIVA-VOCE QUESTIONS AND ANSWERS

## GENERAL

1. **Define Normality (N)**
   It is the number of gram equivalents of the solute dissolved in one $dm^3$ of the solution.
2. **Define Molarity (M)**
   It is the number of moles of the solute dissolved in one $dm^3$ of the solution.
3. **Define Molality (m).**
   It is the number of moles of the solute dissolved in one kg of the solvent.
4. **Define a mole**.
   A mole is the amount of substance, which contains Avogadro number ($6.0122x10^{22}$) of particles.
5. **What is Basicity of an acid?**
   Basicity of an acid is the number of replaceable hydrogen present in one molecules of the acid
6. **Give the Basicity of some common acids?**
   Basicity of HCl, $HNO_3$, and $CH_3COOH$ (acetic acid) is one.
   Basicity of $H_2SO_4$ and $H_2C_2O_4$ (oxalic acid) is two.
   Basicity of $H_3PO_4$ is three.
7. **Why the Basicity of $CH_3COOH$ is one even through three are four hydrogen atoms in one molecule of the acid?**
   The Basicity is one because the number of replaceable hydrogen is only one.[The hydrogen in –COOH group is replaceable; the hydrogen in $–CH_3$ group in not replaceable].
8. **Define the equivalent weight of an acid?**
   $$Equivalent\ weight\ of\ an\ acid = \frac{Molecular\ weight\ of\ acid}{basicity\ of\ the\ acid}$$
9. **Define acidity of a base?**
   Acidity of a base is the number of replaceable hydroxyl group present in one molecule of the base.
10. **Give the acidity of some bases?**

    Acidity of NaOH, KOH and $NH_4OH$ is one.
    Acidity of Ca $(OH)_2$ and $Ba(OH)_2$ is two.
    Acidity of Al $(OH)_3$ is three.
11. **Define Equivalent Weight of a base?**
    $$Equivalent\ weight\ of\ a\ base = \frac{Molecular\ weight\ of\ base}{Acidity\ of\ the\ base}$$
12. **Given (a) Normality and (b) Gram equivalent weight, how can we find out Mass of the Substance per $dm^3$ of solution?**
    Mass of substance per $dm^3$ =Normality x Gram equivalent weight.
13. **How mass of substances per $dm^3$ of solution related to Molarity and gram molecular weight.**
    Mass of substance per $dm^3$ = Molarity x Gram molecular weight.
14. **What are indicators?**
    Indicators are the substances, which indicate the end point in a titration by change of colour.

**15. What are different types of indicators used in volumetric analysis?**

1. Acid base indicators, eg. Phenolphthalein.
2. Metal ion indicators, eg. Eriochrome black-T, Patton and Reader's indicator
3. Redox indicator, eg, Ferroin
4. Internal indicator eg, phenolphthalein, Eriochrome black-T, Patton and Reader's
5. External indicator, eg, potassium ferricynide.
6. Self indicator, eg, Potassium permanganate.

**16. Define the equivalent of an oxidizing?**

It is the number of parts by mass of the oxidizing agent which contains 8 parts by mass of available oxygen for oxidation. [Gram equivalent mass of an oxidizing agent may be defined as the mass of the substance which can accept one mole of electron]

**17. What is a standard solution?**

A standard solution is one, which contains a known mass of the substance dissolved in a known volume of the solution.

## B1: CONDUCTOMETRIC ESTIMATION OF THE ACID MIXTURE

**1. State ohm's law.**

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

**2. What is conductance?**

The reciprocal of resistance is called conductance

**3. What is conductivity?**

The reciprocal of resistivity is called conductivity.

**4. What is the unit of conductance?**

The unit of conductance is ohm$^{-1}$ m$^{-1}$orSm$^{-1}$.

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

A solution may have the following conductance.

1. Specific conductance
2. Equivalent conductance.
3. Molar conductance.

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

The specific conductance is measured.

**7. What is specific conductance?**

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

**8. What is equivalent conductance?**

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

**9. What is molar conductance?**

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

**10. What is cell?**

A device, which produces an electromotive force and delivers an electric current as the function

**11. What is the principle involved in conductometric titration?**

In conductometric titration, there is a sudden change in conductance of a solution near the end point. Hence the end point is determined graphically by plotting conductance against titre

values. The principle underlying conductometric titration is the replacement of ions of a particular conductance by ions of different conductance during the titration.

**12. What factor determines the conductance of a solution?**

Two factors determining the conductance of a solution are

. Mobility of ions- higher the mobility, higher is the conductance and

Number of ions- more the number of ions in solution more is the conductance.

**13. Explain conductometric graph.**

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

## B2: POTENTIOMETRIC ESTIMATION OF FAS USING STANDARD $K_2Cr_2O_7$ SOLUTION

1. **What is Potentiometric titration?**
   The determination of the equivalent point of redox titration on the basis of potential measurement is called a Potentiometric titration.
2. **What are the electrodes used in the determination of FAS potentiometrically?**
   The indicator electrode used is the platinum electrode (acts as anode) and the reference electrode used is the calomel electrode (acts as cathode).
3. **Give the principle of Potentiometric titration**
   The principle 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 titration.
4. **What is an indicator electrode?**
   The electrode whose potential is dependent upon concentration of the ions in solution is termed as indicator electrode. eg: platinum electrode .
5. **What is the full form of FAS?**
   The full form of FAS is ferrous ammonium sulphate.
6. **What is the reaction occurs between FAS and $K_2Cr_2O_7$ in Potentiometric titration?**
   Acidified $K_2Cr_2O_7$ oxidizes ferrous sulphate (FAS) to ferric sulphate. $K_2Cr_2O_7$ itself gets reduced to chromic sulphate.
7. **What is the determining factor in the oxidation- reduction reaction?**

The determining factor is the ratio of the concentration of the oxidation and reduction forms

i.e. $Fe^{2+} \leftrightarrow Fe^{3+} + e$

8. **Why is $H_2SO_4$ added to FAS solution during emf measurement?**
   Reaction between FAS and $K_2Cr_2O_7$ is a redox reaction, where oxidizing agent $K_2Cr_2O_7$ only in the presence of acidic medium oxidizes $Fe^{2+}$ to $Fe^{3+}$.
9. **What is the oxidation state of iron in FAS?**

   It is +2

10. **Why the emf is rises steeply after the equivalent point?**
    This is because, the potential of the solution before the equivalence point is determined $Fe^{2+} \rightarrow Fe^{3+}$ by system only i.e., 0.75V, while at equivalence point, it is determined by both $Fe^{3+}$ and $Cr_2O_7^{2-}$ ions which is = 1.04V. But beyond equivalence point, the potential of the solution is determined by $Cr_2O_7^{2-} / Cr^{3+}$ only i.e. = 1.33V. Therefore, just after the equivalence point, the potential of the solution raises steely.
11. **What is single electrode potential?**
    The potential that is developed when an electrode is in contact with a solution containing its own ions is called single electrode potential.
12. **What is emf?**
    The emf is the potential difference required to drive a current across the electrodes.

## B3: DETERMINATION OF $P^{Ka}$ OF VINEGAR USING $P^{H}$ SENSOR ( GLASS ELECTRODE)

1. **Which acid is present in the vinegar?**
   Acetic acid $CH_3COOH$
2. **What is a weak acid?**
   A weak acid is an acid, which ionizes to a small extent in solution.
3. **Give an example for a weak acid?**
   Acetic acid $CH_3COOH$

4. **What are the ions formed by the dissociation of acetic acid?**
   Dissociation of acetic acid gives acetate ($CH_3COO^-$) ions and hydrogen ions ($H^+$)
   $$CH3COOH \longrightarrow CH_3COO^- + H^+$$
5. **What are strong acids?**
   A strong acid is an acid, which ionizes completely in solution

6. **Give example for strong acids**
   Strong acids: HCl, $HNO_3$, and $H_2SO_4$
7. **What is Ka?**
   Ka is the dissociation constant of weak acid. It is given by the equation

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

8. **What is pKa of weak acid?**

   pKa of weak acid is the negative logarithm to base 10 of dissociation constant of the weak acid . pKa = - $\log_{10}$ Ka
9. **What is pH?**
   pH is the negative logarithm of base 10 of molar concentration of hydrogen ions
   i.e. pH = -$\log_{10}$ ($H^+$)
10. **How are pH & pKa related?**
    They are related by Henderson- Haselbalch equation, which is given by

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

    Where [salt] & [acid] are molar concentration of salt & the acid respectively

11. **What are the electrodes used in the measurement of pH?**
    Glass electrode – Calomel electrode assembly is used in the measurement of pH.
12. **How pH becomes equal to pKa at half equivalence point?**
    At half equivalence point molar concentration of salt is equal to molar concentration of the acid i.e. (salt) = (acid)
    Therefore log (salt)/(acid) = log1= 0
    Thus pH = pKa + 0 = pKa
13. **Explain pH scale**
    pH = 0-7: Acidic: pH = 7-14: Basic; pH = 7: Neutral
14. **Why does pH increase suddenly at the equivalence point?**
    At the equivalence point, the base has neutralized all the weak acid. Afterwards, the concentration of hydroxyl ions increases resulting in a sudden increase of pH

15. **How are Ka & strength of weak acid related?**
    Higher the Ka, stronger is the acid.
16. **How are pKa & strength of weak acid related?**
    Higher the pKa, weaker is the acid.
17. **Why glass electrode called an ion selective electrode?**
    The glass electrode is called an ion selective electrode because, it is able to respond to certain specific ions ($H^+$ ions) only and develop a potential while ignoring the other ions in a solution.

## B4 : ESTIMATION OF TOTAL HARDNESS OF WATER BY EDTA METHOD.

1) **What is hard water?**
   Hard water is water, which does not give lather with soap easily and also, produces scale in hot water pipes, heaters, boilers, etc.
2) **How are the water classified based on the degree of hardness?**

| | |
|---|---|
| Soft water | 0- 75 mg/ liter |

| | |
|---|---|
| Moderately hard water | 75 – 150 mg/liter |
| Hard water | 150-300 mg/liter |
| Very hard water | Greater than 300 mg/liter |

3) **How is hardness of water caused?**
Hardness of water caused by divalent metallic cations in association with anions such as $HCO_3^-$, $SO_4^{2-}$, $Cl^-$ etc. The principle hardness causing cations are calcium and magnesium ions.

4) **How the bicarbonates are introduced into water although Ca (or Mg) is in the insoluble form in the nature?**
Limestone and Dolomite consist of carbonates of calcium and magnesium, which are insoluble water. The rainwater containing CO2 when comes in contact with these carbonates form soluble bicarbonates.

5) **How is hardness in water classified?**
Hardness in water is classified as i). Temporary hardness and ii). Permanent hardness.

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

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

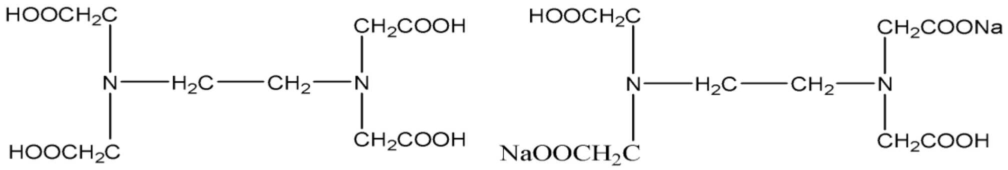
$$Ca(HCO_3)_2 \xrightarrow{\Delta} CaCO_3 \downarrow + H_2O + CO_2$$

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

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

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

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



12) **Why is disodium salt of EDTA preferred to EDTA?**

EDTA is sparingly soluble in water. Its disodium salt is more soluble as it is ionisable and hence it is preferred.

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

14) **What is buffer solution?**

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

15) **Why is ammonia-ammonium chloride buffer added?**

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

16) **What is the chemical name of Eriochrome Black T?**

The chemical name of Eriochrome Black-T is sodium 1- (1- hydroxyl-2-naphthylazo)-6-nitro-2-naohthol-4-sulphaonate SHNNNS).

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

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

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

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

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

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

18) **Why are the titrations involving EDTA carried out slowly towards the end point?**

The titrations involving EDTA are carried out slowly towards the end point, because, the rate of formation of the metal complex of EDTA is very slow.

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

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

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

## C1: ESTIMATION OF COPPER PRESENT IN ELECTROPLATING EFFLUENT BY OPTICAL SENSOR (COLORIMETRY)

1. **What is Colorimetry?**

   Chemical analysis through measurement of absorption of light in the visible region is known as Colorimetry.

2. **What is the range of wavelengths of light in the visible region?**

   400 nm to 700 nm. (nm = nano meter = $10^{-9}$ meter)

3. **What forms the basis for the colorimetric determination?**

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

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

5. **What is the basic principle of colorimetric measurements?**

It consists of comparing under well defined conditions, the colour produce by the substance in unknown amount with the same colour produced by a known amount of the material being determined.

6. **Why filters are used in colorimetric experiment?**
   Filters are used for selecting desired spectral region

7. **What is wavelength?**
   The distance between any two successive peaks or troughs of wave is the called wavelength. It is represented by $\lambda$. It can be expressed meters.

8. **What is wave number?**
   Wave number is reciprocal of wavelength. It is the number of waves present in one meter length. It can be expressed in $m^{-1}$

9. **What is the wavelength at which copper estimation is done?**
   At 620 nm

10. **What is frequency?**
    It is the number of waves passing through a point second. It is represented by "ν" It is expressed in Hz.

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

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

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

$$A = \epsilon cl$$

Where, $\epsilon$ = molar extinction coefficient.

14. **What is a calibration curve?**
    It is the plot of absorbance against concentration of solutions. For solutions obeying Beer's law, this is a straight line.

15. **What is transmittance?**
    It is the ratio of the intensity of transmitted light ($I_t$) to that of the incident light ($I_o$). $I_t / I_o = T$
16. **Why are different volumes of solution taken in the flasks?**

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

17. **What is blank solution?**
    A blank solution is identical in all respects to test solution except for the absence of test solute.
18. **Why is blank solution used in colorimetric estimation?**
    To nullify absorbance caused due to the coloring impurities present in the reagents.

19. **Why is ammonia added? Why is the same amount of ammonia added to different volume of $CuSO_4$ solution?**
    Ammonia is added to get cuprammonium sulphate, $[Cu (NH_3)] SO_4$, a dark blue complex. Same amount ammonia is added to nullify the absorbance due to any coloring impurities present in ammonia.
20. **Why is estimation of copper done at 620nm wavelength?**
    The estimation of copper is carried at 620nm wavelength because; the complex shows a maximum absorbance at 620nm.

## C2: DETERMINATION OF VISCOSITY CO-EFFICIENT OF LUBRICANT 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 co-efficient of a liquid?**
   The viscosity co-efficient of a liquid is defined as the tangential force per unit area required maintaining a unit velocity gradient between any two successive layers of a liquid situated unit distance apart.
3. **What is density of liquid?**
   The density of a liquid is its mass/ its volume.
4. **The density of a substance is expressed relative to what?**
   The density of a substance is expressed relatively to that of water at $4^oC$.
5. **What is SI unit of viscosity co-efficient?**
   The viscosity co-efficient is expressed as kg/m/s.
6. **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.
7. **What are the factors that influence the viscosity of a liquid?**
   1.Increase in molecular weight results in an increase in viscosity.

2. Branched chain compounds have higher viscosity than those involving straight chain compounds.
3. The polar compounds are more viscous than the polar ones. The presence of hydrogen bonds causes the viscosity to increase.

4. Temperature has marked influences by the viscosity of a liquid.

8. **What is the law base on the viscous flow of liquid through capillary tubes?**

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

   Where η = Viscosity co-efficient; p = Hydrostatic pressure; r = Radius of the tubes; t = Time required for the volume; v = of the liquid to flow through the tube of the length, l.
9. **How does the viscosity vary with temperature?**
   The viscosity of a liquid usually decreases with the rise of temperature.
10. **Why should the viscometer be dried before the measurements are done?**
   The viscometer should be dried to avoid formation of an emulsion, which changes the rate of flow of the liquid.
11. **Why acetone used for cleaning viscometer?**
   Acetone is a volatile organic liquid. To dry the viscometer quickly, it is rinsed with acetone.
12. **Why viscometer not rinsed with the given liquid or water?**
   If the viscometer is rinsed with the given liquid or water before measuring the flow times the volume taken will be more than a definite known volume.
13. **Why do require laboratory temperature for viscosity determination?**
   Because, the physical constants like density and viscosity of a liquid vary with temperature.
14. **How is the viscosity of a liquid related to its mobility?**
   Viscosity of a liquid is inversely proportional to its mobility.
15. **Why is viscometer dipped in water bath?**
   Viscometer is dipped in water bath to maintain constant temperature.

## C3 : ESTIMATION OF PERCENTAGE OF IRON IN TMT BAR BY EXTERNAL INDICATOR METHOD

1. **What is the role of stannous chloride ($SnCl_2$) in the determination of iron in haematite ore?**
   Stannous chloride acts as a reducing agent. It reduces $Fe^{3+}$ to $Fe^{2+}$ i.e. ferric to ferrous.
   $2FeCl_3 + SnCl_2 \rightarrow 2FeCl_2 + SnCl_4$.
2. **What happens to stannous chloride in the above reaction?**

   Stannous chloride (2+) is oxidized to stannic chloride (4+).

3. **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 the titration get reduced to ferrous ions. As a result, the volume of titrant, potassium dichromate consumed will be more

4. **Why is mercuric chloride added?**

   Mercuric chloride is added to remove excess stannous chloride. Mercuric chloride oxidizes stannous chloride to stannic chloride. It gets reduced to mercurous chloride.

   $SnCl_2$ (2+) + $2HgCl_2$ (2+) → $Hg_2Cl_2$ (1+) + $SnCl_4$ (4+).

5. **Is mercurous chloride soluble in water?**

   No. It is not soluble. That is why it appears as a precipitate, which is silky white in colour.

6. **What is the oxidation of tin is stannous chloride ($SnCl_2$)?**

   2+

7. **What is the oxidation state of tin is stannic chloride?**

   4+

8. **What is the oxidation state of mercury is mercuric chloride ($HgCl_2$)?**

   2+

9. **What is the oxidation state of mercury in mercurous chloride ($Hg_2Cl_2$)?**

   1+

10. **Which is indicator used in the determination of iron in haematite ore?**

    Potassium ferricynide, [$K_3Fe$ $(CN)_6$], is used as an external indicator.

11. **What is the colour of [$K_3Fe$ $(CN)_6$] with ferrous ion?**

    Deep blue colour.

12. **What is the compound with deep blue colour?**

    Ferrous ferricynide complex.

13. **Why the colour of indicator drops is remains unchanged at the end point?**

    Only ferrous ions form a blue colour with potassium ferricynide. Since there are no $Fe^{2+}$ ions left in the conical flask at the end point, no blue colour is formed. In the conical flask, at the end point, $Fe^{3+}$ ions are present, but they do not give colour with potassium ferricynide.

14. **What happens to $Fe^{2+}$ when you titrate $Fe^{2+}$ solution with $K_2Cr_2O_7$?**

    $Fe^{2+}$ is oxidized to $Fe^{3+}$ by potassium dichromate.

15. **What is the reaction that occurs during titration?**

    Acidified potassium dichromate oxidizes ferrous ions present in the haematite ore solution to ferric state. It itself gets reduced to chromic ion.

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

16. **Why is potassium ferricynide not used as an internal indicator?**

    Potassium ferricynide can not be used as an internal indicator in the determination of iron in haematite because, potassium ferricynide combines irreversibly with ferrous ion to form a deep blue ferrous ferricynide (Turnbull's) These ferrous ions involved in complex formation are not available for reaction with potassium dichromate. Moreover, the end point cannot be detected, as there is no colour change.

    $$3Fe^{2+} + 2K_2\,[Fe(CN)_6] \rightarrow Fe_3[Fe(CN)_6]_2 + 4K^+$$

17. **Why can't we use potassium ferrocyanide as an indicator?**

    Potassium ferrocynide does not react with ferrous ions.

## C4: DETERMINATION OF COD OF INDUSTRIAL WASTE WATER SAMPLE

1. **What is meant by industrial sewage?**

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

2. **What is the full form of COD?**

   COD stands for chemical oxygen demand.

3. **What is chemical oxygen demand?**

   It is the amount of oxygen required for oxidation of biologically and non-biologically oxidisable organic compound present in 1dm$^3$ of waste water by a strong oxidizing agent such as acidified potassium dichromate.

4. **What is full form of BOD?**

   BOD stands for biological oxygen demand.

5. **What is biological oxygen demand?**

   It is the amount of oxygen requires for the biological oxidation of organic compounds present in 1dm$^3$ of wastewater over a 5 days period at 20 $^o$C.

6. **What is the role of silver sulphate in the determination of COD?**

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

7. **What is the role of mercuric sulphate in the determination of COD?**

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

8. **What are the oxidisable impurities present in waste water?**

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

9. **What is the indicator used in COD experiment?**

   Ferroin indicator (Ferrous 1, 10 phenanthroline sulphate)

10. **What is the colour at the end point?**

    Blue green to reddish brown.

**11. Why the sulphuric acid is added during the preparation of standard FAS solution?**

Sulphuric acid is added to prevent the hydrolysis of ferrous sulphate into ferrous hydroxide.

**12. What is the role of $K_2Cr_2O_7$ in COD analysis?**

Potassium dichromate oxidizes the organic matter present in waste water.

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

$K_2Cr_2O_7$ oxidizes organic matter to $CO_2$ and $H_2O$

**14. Which organic compounds are not oxidized in COD analysis?**

Aromatic hydrocarbons and pyridine are not oxidized in COD test.

**15. Mention a few application of COD analysis?**

The COD test is used in analysis of industrial waste water.

The COD test gives the pollution level of industrial waste water.

**16. Among COD and BOD, which is higher in value?**

COD is higher in value

**17. What is the unit in which COD is expressed?**

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