Laboratory Manual

for

Engineering Chemistry Laboratory (BSC106B)

B.Tech. 1st Semester

Department of Chemistry

Name of the student	
University Reg. No	

Faculty of Science and Humanities

Ramaiah University of Applied Sciences

University House, New BEL Road, MSR Nagar, Bangalore – 560054

www.msruas.ac.in

Engineering Chemistry Laboratory

Faculty	Science and Humanities
Program	B. Tech
Branch	Computer Engineering / Civil Engineering /Electrical Engineering / Electronics and Communication Engineering / Mechanical Engineering / Aerospace Engineering / Automotive Engineering
Year/semester	1st Year/1st and 2nd Semester
Department	Chemistry
Course	Engineering Chemistry Laboratory
Course code	BSC106B

Instructions for Engineering Chemistry Laboratory

- The objective of the laboratory is **learning**. The experiments are designed to illustrate phenomena in different areas of chemical kinetic study, electrochemistry, metallurgy, polymers and to expose to the measuring instruments.
- Conduct the experiments with interest and an attitude of learning.
- Come well prepared for the experiment.
- Work quietly and carefully (the whole purpose of conducting experiments is to make reliable measurement) and equally share the work with your partners.
- Be honest in recording and representing your data.
- All presentations of data, tables and graphs calculations should be neatly and carefully done.
- Bring necessary graph papers for each experiment. Learn to optimize on usage of graph papers.
- Graphs should be neatly drawn with pencil.
- Come equipped with calculator, scales, pencils etc.
- Do not handle idly with apparatus. Manage instruments with care.
- Check the labels on the reagent bottles before using.
- Do not waste reagents unnecessarily.
- Read the first aid measures displayed in the laboratory and follow them in case of exposure to hazardous chemical reagents.
- Take necessary precautions and safety measures before conducting the experiment.

LABORATORY REPORT

Course Code: BSC106B

Course Name: Engineering Chemistry Laboratory

Programme: B.Tech. (All Branches)

Department: Chemistry

Faculty: Faculty of Science and Humanities

Name of the Student:

Register Number:

Semester / Year:

Course Leader(s):

Ramaiah University of Applied Sciences

470 P, IV phase, Peenya Industrial Area, Bengaluru, Karnataka,

INDIA - 560 058

Declaration sheet

Student Name			
Register Number			
Programme/Course	B.Tech. / Engineering Chemistry	Semester/Year	1/2017
Course Code	BSC106A		
Course Title	Engineering Chemistry Laboratory		
Course Date		To	
Course Leaders			

Declaration

The laboratory report submitted herewith is a result of my own investigations and that I have conformed to the guidelines against plagiarism as laid out in the Student Handbook. All sections of the text and results, which have been obtained from other source, are fully referenced. I understand that cheating and plagiarism constitute a breach of university regulations and will be dealt with accordingly.

Signature of the Student		Date	
Submission date stamp			
Signature of the Course Leader and date			
Final Marks Obtained	**Remarks**		

Name: Reg. Number:

Index sheet

Experiment No	Experiment Name	Page No	Marks Obtained
01	Determination of viscosity coefficient of a given liquid using Ostwald's viscometer		
02	Conductometric estimation of an Acid using standard NaOH solution		
03	Potentiometric estimation of FAS using standard $K_2Cr_2O_7$ solution		
04	Colorimetric estimation of nickel using Nickel Dimethylglyoxime Complex		
05	Determination of first order reaction constant for acid hydrolysis of ethyl acetate		
06	Determination of percentage of iron from the given iron ore sample		
07	Determination of efficiency of electroplating of copper		
08	Anodizing of aluminium and measurement of its thickness		
09	Colorimetric estimation of copper and zinc present in a given brass sample		
10	Measurement of voltage in a hydrogen-oxygen fuel cell*		
11	Determination of calorific value of a fuel sample using Bomb calorimeter*		
12	Demonstration of producing printed circuit board*		
13	Preparation of a polymer composite by hand layup technique*		
14	Construction and operation of lead acid battery cell*		
15	Determination of empirical formula of a fuel using exhaust gas analyser*		
Total Marks			

* Demonstration Experiments

Title of the Laboratory Exercise: Determination of relative viscosity coefficient of a given liquid using Ostwald's viscometer

1. **Introduction**:

   Viscosity is the internal friction or resistance of a substance during flow. Viscosity of liquids depends on many factors such as, intermolecular attractive forces, molecular weight, structure and shape of molecules, temperature, pressure, etc. viscosity data helps us to understand the flow behaviour of liquids while designing the sewer line, pumps, pipes etc and also helps in selecting good lubricating oil. A slow moving layer exerts a friction on its nearest moving layer. Coefficient of viscosity or absolute viscosity coefficient, μ, is defined as the force per unit area necessary to maintain a unit velocity gradient between two parallel layers of liquid kept at unit distance apart. The CGS unit of viscosity is Poise (P). The numerical values are usually given in smaller subsidiary unit such as, the millipoise (mP = 10^{-3} P) or the centipoises (cP = 10^{-2} P). The SI unit of viscosity is Newton.s.m^{-2} (or kg s^{-1} m^{-1}). Viscosity of liquids is measured by studying their streamlined flow, through capillary tube and using Poiseuille's formula:

$$\mu = \frac{\pi r^4 P t}{8 V l}$$

   where μ, is the absolute coefficient of viscosity, P is hydrostatic pressure responsible for the flow, r and l are the radius and length of the capillary tube in centimeter, and V is the volume of the liquid in cubic centimeter flowing in t seconds. Pressure, P is given by hdg where, h is the height of the liquid causing flow, d is its density and g is acceleration due to gravity.

   Taking a standard liquid (say, distilled water) of known coefficient η_w of viscosity, the coefficient of viscosity, η_l of test liquid can be calculated by measuring flow times for equal volumes of the standard liquid and the test liquid through the same capillary at the specified temperature.

   Relative Viscosity, $\dfrac{\mu_1}{\mu_w} = \dfrac{t_1\, \rho_1}{t_w\, \rho_w}$, Absolute viscosity, $\mu_1 = \mu_w \dfrac{t_1\, \rho_1}{t_w\, \rho_w}$

   where t_l, t_w are flow times and ρ_l, ρ_w are the densities of the test liquid and distilled water respectively. Knowing the densities of water and test liquid and viscosity of distilled water at experimental temperature, the absolute viscosity of the test liquid can be calculated.

Significance of Viscosity:

Knowledge of viscosity of a liquid is essential in chemical equipment design such as chemical reactors, mixers, etc. Viscosity of liquid has a direct and large effect on heat transfer coefficients, which are especially important for design of heat exchangers and various other heat transfer equipment. Viscosity data are essential to understand flow behavior of liquids in designing of the sewer line, pumps, pipes, etc. Knowing viscosity helps in selecting good lubricating oil as highly viscous liquids can carry heavy loads at high temperatures.

2. **Aim and Objectives:**
    Aim: To determine Relative Viscosity coefficient of given unknown liquid

    Objective: Determination of relative viscosity coefficient of a given liquid using Ostwald's viscometer

3. **Experimental Set Up**: Fix an Ostwald's viscometer vertically in a thermostat.
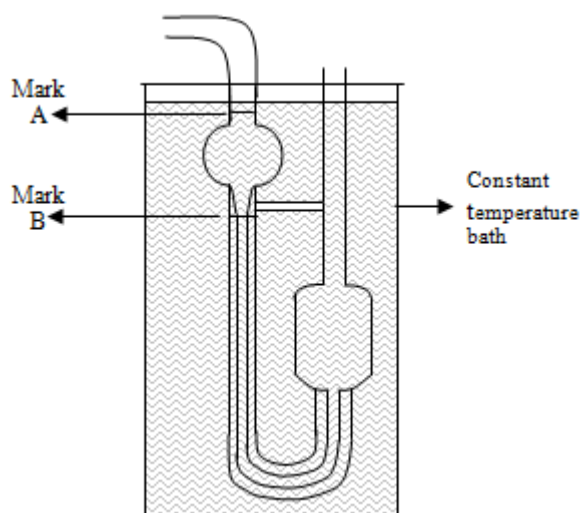
4. **Experimental Procedure:**

    1. Pipette out 10 ml of the given liquid into wider limb of viscometer.
    2. Suck it up in the capillary limb slightly higher than the upper mark above the glass bulb.
    3. Start a stop-watch when the lower meniscus of liquid crosses the upper mark (above the bulb) and stop it when the lower meniscus crosses the lower mark (below the bulb). Record the flow time.
    4. Repeat the measurements for concordant values (3times).
    5. Wash and rinse the viscometer with acetone and dry it in a hot air oven.
    6. Cool the viscometer thoroughly and fix it vertically in the thermostat.
    7. Take identical volume (10 ml) of distilled water in the viscometer and measure its flow time.

5. **Data Collection and Tabulation:** Temperature of the thermostat   = …………. °C

Flow time measurements:

Liquid	Flow time (seconds)			
	Trial - 1	Trial - 2	Trial - 3	Average (s)
Test liquid				t_l =
Water				t_w =

6. **Calculations**: Relative viscosity coefficient of the test liquid, $\mu_l = \mu_w \dfrac{t_l \, \rho_l}{t_w \, \rho_w}$

Time of flow of test liquid $\quad\quad t_l$ = ………….. s

Density of test liquid $\quad\quad\quad\quad \rho_l$ = ………….. g/cm^3

Time of flow of water $\quad\quad\quad\quad t_w$ = ………….. s

Density of water $\quad\quad\quad\quad\quad\quad \rho_w$ = ………….. g/cm^3

Viscosity coefficient of water $\quad\quad \mu_w$ = ………….. Nsm^{-2} (Pa s)

7. **Result**: ………….. Nsm^{-2} (Pa s)

8. **Conclusions:**

9. **Comments:**

      a. Limitations of Experiments

      b. Limitations of Results

      c. Learning happened

      d. Recommendations

Assessment:

Short Procedure	(2.5 marks)	
Performance/ Observation	(12 marks)	
Calculation + Graph	(3 marks)	
Viva-voce	(7.5 marks)	
Total	(25 marks)	

Title of the Laboratory Exercise: Conductometric estimation of an acid using standard NaOH solution

1. **Introduction:**

   Conductance of any solution (C) is the ease with which current flows through it. It is the reciprocal of resistance (R) offered by the solution.

   $$C = 1/R$$

   Conductance of a solution depends on number of ions, charge on the ions and also on the mobility of the ions. During the course of a titration, the conductance of the solution also changes. Hence, there is a sharp break in the conductance - volume (of titrant) curves. This break corresponds to the equivalence point. Such titrations in which conductance measurements are made use of in determining the equivalence point are called conductometric titrations. *The principle behind this experiment is replacement of ions of a particular conductance by ions of different conductance.*

   Among all conductometric titrations, reaction involving strong acid (HCl) and strong base (NaOH) is the most common activity.

   Hydrochloric acid is a strong electrolyte and would be completely in dissociated state. The conductance of this solution is the maximum, before the addition of NaOH. Upon addition of NaOH, highly mobile H^+ ions (350 mho.cm^2.mol^{-1}) of the acid are replaced by less mobile Na^+ ions (48 mho.cm^2.mol^{-1}).

   $$H^+ + Cl^- + Na^+ + OH^- \longrightarrow Na^+ + Cl^- + H_2O$$
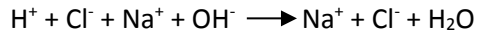
   Hence the conductance would decrease steeply. The same trend would continue till all the H^+ ions available in the acid are completely reacted with the added NaOH. Now, the addition of NaOH would add more and more of Na^+ and OH^- ions. Due to relatively higher mobility of OH^- ions (193 mho.cm^2.mol^{-1}), the conductance of the solution increases again.

   Advantages of Conductometric Titrations:

   Mixture of acids can be titrated more accurately by conductometric titration. Conductometric titrations may be applied where potentiometric methods fail. They give accurate results in dilute solution as well as in concentrated solution. It can be employed with colored solutions. Very weak acids which cannot be titrated potentiometrically in aqueous solutions can be titrated conductometrically with relative ease.

2. **Aim and Objectives**

   Aim: To estimate the concentration of hydrochloric acid

   Objectives: To find concentration of HCl present in 1000 cm^3 using conductometer

3. **Apparatus:** Pipette, burette, conductometer, conductivity cell and a beaker

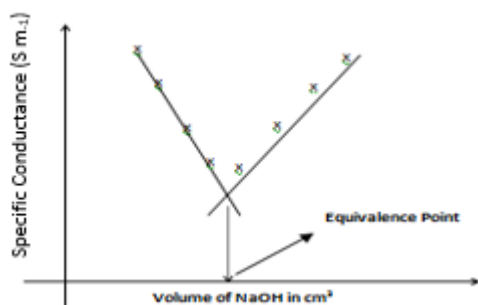4. **Experimental Procedure**
   a. Pipette out 25 cm^3 of acid into a clean 100 cm^3 beaker.

b. Dip the conductivity cell in the acid, such that the two platinum electrodes are immersed completely.
c. The cell is connected to the conductivity bridge.
d. Measure the specific conductance at 0 volume of NaOH.
e. Take the given NaOH solution in a burette rinsed with the same solution.
f. Rundown 0.5 cm³ of NaOH solution each time into the beaker and shake well (using a glass road). Note down the specific conductance.
g. Specific conductance decreases in the beginning, increases slightly and finally starts increasing by large value.
h. Take around 8-10 readings on the increasing side.
i. Plot a graph of specific conductance versus volume of sodium hydroxide added.
j. Determine the volume of sodium hydroxide required to neutralize HCl.
k. From the normality of sodium hydroxide, calculate the normality and amount of HCl

5. **Data Collection and Tabulation**

Volume of NaOH in cm³	Specific Conductance (S m⁻¹)
0.0	
0.5	
1.0	
1.5	
2.0	
2.5	
3.0	
3.5	
4.0	
4.5	
5.0	
5.5	
6.0	
6.5	
7.0	
7.5	
8.0	
8.5	
9.0	

Nature of graph

6. **Calculations:**

$$(NV)_{HCl} = (NV)_{NaOH}$$

$$N_{HCl} = \frac{(NV)_{NAOH}}{V_{HC1}}$$

Amount of HCl present in 1000 cm^3 of its solution $= N_{HCl}$ x gram equivalent weight of HCl (36.5)

$$=$$

7. **Result:** Concentration of HCl $=$ ………………….g/lit

8. **Conclusions**
9. **Comments**

   a. Limitations of Experiments

   b. Limitations of Results

   c. Learning happened

   d. Recommendations

Assessment:

Short Procedure	(2.5 marks)	
Performance/ Observation	(12 marks)	
Calculation + Graph	(3 marks)	
Viva-voce	(7.5 marks)	
Total	(25 marks)	

Date:

Title of the Laboratory Exercise: Potentiometric estimation of FAS using standard $K_2Cr_2O_7$ solution.

1. **Introduction**:

   Redox titrations can be carried out potentiometrically using platinum-calomel electrode combination. For the reaction,

   Reduced form $\rightarrow$ Oxidized form + n electrons

   The potential is given by Nernst equation,

   $$E = E^0 + \frac{0.0591}{n} \log \frac{[\text{Oxidized form}]}{[\text{Reduced form}]}$$

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

   The reaction that takes place in the determination of Fe^{2+} is

   $Fe^{2+} \rightarrow Fe^{3+} + e$

   $Cr_2O_7^{2-} + 14H^+ + 6e \rightarrow 2Cr^{3+} + 7H_2O$
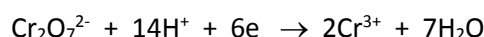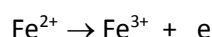
   Prior to the equivalence point, the potential is determined by the Fe^{3+}/Fe^{2+} system and the potential is given by the equation,

   $$E_{cell} = E_{Fe^{2+}} = E^0 + \frac{0.0591}{1} \log \frac{[Fe^{3+}]}{[Fe^{2+}]}$$

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

   The potential of the solution will be around 0.75 V (since the contribution to the potential by the second term is negligible). At the equivalence point the potential is determined by both $E^0{}_{Fe^{2+}}$ and $E^0 Cr_2O_7^{2-}$ and is given by

   $$E_{cell} = \frac{E^0_{Fe^{2+}} + E^0_{Cr_2O_7^{2-}}}{2} = \frac{0.75\text{ v} + 1.33\text{ V}}{2} = 1.04\text{ V}$$

   Beyond the equivalence point the potential is determined by $Cr_2O_7^{2-}/Cr^{3+}$ system given by the equation,

   $$E_{cell} = E_{Cr} = E^0_{\frac{Cr_2O_7^{2-}}{Cr^{3+}}} + \frac{0.0591}{6} \log \frac{[Cr_2O_7^{2-}]}{[Cr^{3+}]}$$

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

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

2. **Aim and Objectives:**

Aim: To estimate the unknown amount of FAS in a given solution using standard $K_2Cr_2O_7$ solution.

Objective: To estimate the amount of FAS in the given FAS solution using $K_2Cr_2O_7$ solution and potentiometer.

3. **Apparatus:** Potentiometer, platinum and calomel electrode, pipette, burette and a beaker.
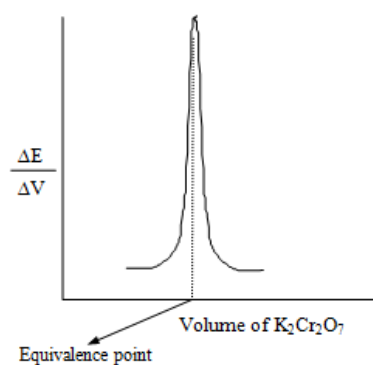
4. **Experimental Procedure:**

   1. Pipette out 25.0 cm^3 of ferrous ammonium sulphate solution into a beaker.
   2. Add half test tube-full of dilute sulphuric acid.
   3. Immerse the Platinum-calomel electrode assembly into the given FAS   solution and connect the electrodes to a potentiometer.
   4. Fill the burette with standard potassium dichromate solution.
   5. Record the potentiometer reading at 0 volume of $K_2Cr_2O_7$ solution.
   6. Add 0.5 cm^3 of $K_2Cr_2O_7$ to the beaker. Stir the solution carefully and measure the potential after 15 seconds.
   7. Continue the procedure till the potential shows a tendency to increase rapidly. Further, take six readings.
   8. Determine the end point by differential method i.e., by plotting $\Delta E/\Delta V$ against volume of potassium dichromate as shown in the figure.
   9. Calculate the normality of the ferrous ammonium sulphate solution and determine the amount of iron in the given solution.

5. **Data Collection and Tabulation:**

Volume of $K_2Cr_2O_7$ (ml)	E (mV)	ΔE (mV)	ΔV (ml)	$\dfrac{\Delta E}{\Delta V}$ (V/L)
0		---------	---------	---------
0.5				
1.0				
1.5				
2.0				
2.5				
3.0				
3.5				
4.0				
4.5				
5.0				
5.5				
6.0				
6.5				

7.0				
7.5				
8.0				
8.5				
9.0				
9.5				
10.0				

Nature of the graph:

$\frac{\Delta E}{\Delta V}$

Volume of $K_2Cr_2O_7$

Equivalence point

6.   Calculations:

From the graph: …… V cm³of $K_2Cr_2O_7$ is required to oxidize   25 cm³ FAS.

$$N_{FAS} = \frac{N_{K2Cr2O7} \times V_{K2Cr2O7}}{V_{FAS}} = \text{…………} = \qquad N$$

Quantity of FAS in 1 dm³ of the given solution $= N_{FAS} \times$ equivalent weight of FAS

$$= \quad \text{………………} \qquad g$$

7. Result: The concentration of FAS   =   ………………g/lit

9. Conclusions:

10. Comments:

a. Limitations of Experiments

b. Limitations of Results

c. Learning happened

d. Recommendations

Assessment:

Short Procedure	(2.5 marks)	
Performance/ Observation	(12 marks)	
Calculation + Graph	(3 marks)	
Viva-voce	(7.5 marks)	
Total	(25 marks)	

Title of the Laboratory Exercise: Colorimetric estimation of Nickel using Nickel Dimethylglyoxime complex

1. **Introduction:**

Colorimetric analysis depends upon the measurement of quantity of light absorbed by a coloured solution. Quantitative measurement of absorption is based on Beer-Lambert's law, which states that, the amount of light absorbed by coloured solution is proportional to the thickness and concentration of the light absorbing medium. It can be expressed as

Absorbance (or optical density, O.D.), $\log\left(\dfrac{I_0}{I_t}\right) = \varepsilon c t$

where, 'I_0' is intensity of incident light, 'I_t' is intensity of transmitted light, 't' is thickness of the light absorbing medium (cm), 'c' is concentration of the coloured constituent in the solution (mol/L) and 'ε' is molar extinction coefficient (amount of light absorbed when 't' and 'c' are 1 cm and 1 mol/L respectively). Since, ε is a constant, absorbance is proportional to the concentration of coloured constituent in solution, provided the thickness of the light absorbing medium is kept constant.

Significance of Colorimetry: The data obtained from colorimeter will be helpful to adjust the colour components in food industry and also to monitor the growth of a bacterial or yeast culture. In laboratories, it is used to test water quality for ions such as Ni^{2+}, iron, molybdenum, etc. and also to determine the concentrations of plant nutrients.

Principle: Nickel (200-400 *J.lg)* forms the red Nickel-dimethylglyoxime complex in a slightly alkaline/Acidic medium. Reliability of colorimetric analysis requires appreciable colour intensity of solution. Therefore, light blue coloured Ni^{2+} is reacted with dimethylglyoxime to give intense red coloured Nickel-dimethylglyoxime complex, $C_8H_{14}N_4NiO_4$. The solution absorbs light of 560 nm wavelength to a maximum extent. A series of standard solutions of nickel complexes is used to generate a calibration curve on a graph of absorbance versus concentration of nickel. Absorbance of the test solution complexed with dimethyl glyoxime is measured and compared on the standard curve for concentration. Thus, concentration of nickel in the test solution is determined.

2. **Aim and Objectives:**

Aim: To determine the amount of nickel present in given unknown solution

Objective: Determination of nickel present in unknown solution using colorimeter

3. **Apparatus:** Colorimeter, cuvette, six 50 ml volumetric flasks, burette and a beaker

4. Experimental Procedure:

**Preparation of stock solution:**

Take given quantity of Nickel sulphate in 100ml standard flask. Add 2ml of sulphuric acid, 4 ml of sodium potassium tartrate and 16ml of potassium per sulphate.

1. To above stock solution add 2 ml of Dimethylglyoxime (DMG) and 5 ml of NaOH, keep it aside for half an hour, then make it up to the mark using distilled water.
2. Take 5ml, 10ml, 15ml, 20ml and 25ml of the stock solution into 5 different 50 ml volumetric flasks and a test solution in another volumetric flask.
3. Dilute with distilled water up to the mark and shake well.
4. Prepare a blank solution by taking all the reagents except nickel sulphate.
5. Place the blank solution in the quartz cell (cuvette) of the instrument and set the absorbance to read zero at 560 nm.
6. Measure the absorbance of the standard solutions and test solution with reference to the blank at 560 nm wavelength.
7. Plot a graph of absorbance Vs Volume of NiDMG Complex, find the test solution volume and calculate the quantity of Ni present in test solution.
8. Draw a calibration curve by plotting the absorbance (y-axis) against volume of NiDMG Complex solution (x-axis).
9. From the calibration curve, find out the volume of the given test solution and calculate the amount of Nickel.

5. Data Collection and Tabulation

Volume of Nickel Dimethylglyoxime (ml)	Absorbance
5	
10	
15	
20	
25	
Test solution (given)	

Graph:
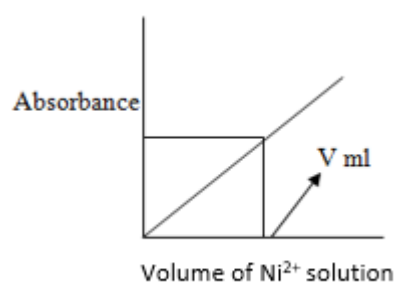
6. Calculations

$1 cm^3$ of the stock solution contains = ------a-----mg of $(NH_4)_2Ni(SO_4)_2 \cdot 6H_2O$

{1 molecule of $(NH_4)_2Ni(SO_4)_2 \cdot 6H_2O \equiv$ 1 atom of Ni)

394.99 mg of $(NH_4)_2Ni(SO_4)_2 \cdot 6H_2O \equiv$ 58.69mg of Ni

a-------- mg of $(NH_4)_2Ni(SO_4)_2 \cdot 6H_2O \equiv \dfrac{58.69 \ x \ -------}{394.99}$ = mg of Ni

$1 cm^3$ of the stock solution contains = b -----------mg of Ni

v----- ------cm^3 of the test solution contains = b ------- x v ---- mg of Ni

= mg of Ni

7. Result: The quantity of nickel in the test solution is ……………mg

8. Conclusions

9. Comments

a. Limitations of Experiments

b. Limitations of Results

c. Learning happened

d. Recommendations

Assessment:

Short Procedure	(2.5 marks)	
Performance/ Observation	(12 marks)	
Calculation + Graph	(3 marks)	
Viva-voce	(7.5 marks)	
Total	(25 marks)	

Title of the Laboratory Exercise: Determination of first order reaction rate constant for acid hydrolysis of ethyl acetate
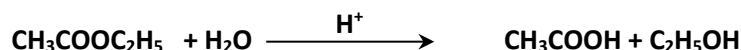
1. Aim and Objectives

Aim: To determine the first order reaction rate constant for acid hydrolysis of ethyl acetate

Objective: Determination of the first order reaction rate constant for acid hydrolysis of ethyl acetate using HCl

2. Apparatus: Burette, pipette, conical flask, water bath, funnel etc

3. Introduction:

$$CH_3COOC_2H_5 \ + H_2O \ \xrightarrow{\quad H^+ \quad} \ CH_3COOH + C_2H_5OH$$

The hydrolysis of ethyl acetate is a first order reaction as rate of reaction is proportional to first power of [ethyl acetate]. Water is in large excess; since there is no appreciable change in its concentration, progress of the reaction is monitored by measuring increase in [CH_3COOH] by titrating against standard NaOH. The velocity constant is calculated by using integrated rate equation.

$$k = \frac{2.303}{t} \log \frac{a}{a-x}$$

Where 'a' is initial concentration and 'a-x' is concentration at time 't'

Significance of the Chemical Kinetic Measurements: Chemical reactors used in various industrial process are designed based on the fundamentals of chemical kinetics. Kinetic measurements are important in drug delivery systems. Principles of Chemical kinetics are widely used in food science. Kinetic modeling is also useful for the nutritionist, to study the kinetics of changes during digestion and assimilation in the body (bioavailability). It helps in the study of rate of diffusion of harmful chemicals expelled by industry, automobiles that are causes of ozone depletion

4. Experimental Procedure:

In a clean and dry conical flask, take 5 cm^3 ethyl acetate and in another, 100 cm^3 0.5N HCl solution. Keep them in water bath to attain room temperature for 5 minutes.

1. In a clean burette, fill 0.1N NaOH after rinsing. Take 2-3 ice pieces and one or two drops of phenolphthalein indicator in a conical flask.
2. Mix ethyl acetate to 0.5N HCl, shake well, pipette out 5 cm^3 of this to conical flask containing ice pieces and indicator. Titrate immediately with 0.1N NaOH till pale pink colour persists. Note down the burette reading as zero minute reading (V_0).
3. Titrate, **5** cm^3 of the same reaction mixture at every **10 minutes** till 30 minutes.

4. The reaction is accelerated to completion by heating to 70°C for ½ hr. During this time the flask must be stoppered to avoid changes in concentration due to evaporation. Cool to room temperature, pipette 5.0 cm^3 and add to 25 cm^3 of water and titrate as before.
5. Calculate the 'k' using given formula.
6. Plot a graph of log (a-x) vs time and calculate k from slope.

Nature of Graph:

$$\text{Slope} = \frac{AB}{BC} = - \frac{K}{2.303}$$
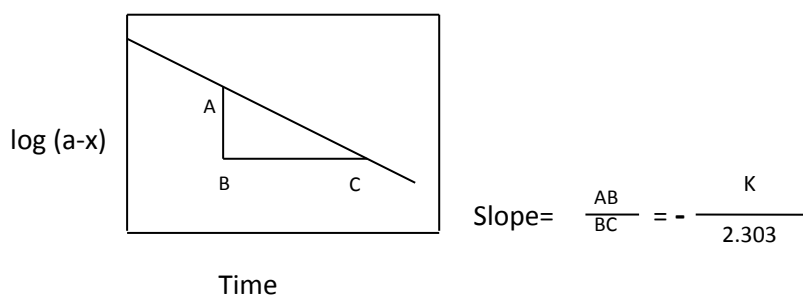
5. Burette Readings

Time (min)	Vol of NaOH (cm^3)	$a = V_\infty - V_o$	$a-x = V_\infty - V_t$	log (a-x)	k (min^{-1})
0	$V_o =$				
10	$V_t =$				
20	$V_t =$				
30	$V_t =$				
∞	$V_\infty =$				

6. Calculation :

$$k = \frac{2.303}{t} \log \frac{a}{a-x}$$

$$\therefore k = \text{slope} \times 2.303 = \dots \quad \text{min}^{-1}$$

7. Result: a. Reaction velocity constant k for Set I by formula method = **min^{-1}**

b. Reaction velocity constant k for Set I by graphical method = **min^{-1}**

8. Conclusions

9. Comments

a. Limitations of Experiments

b. Limitations of Results

c. Learning happened

d. Recommendations

Assessment:

Short Procedure	(2.5 marks)	
Performance/ Observation	(12 marks)	
Calculation + Graph	(3 marks)	
Viva-voce	(7.5 marks)	
Total	(25 marks)	

Title of the Laboratory Exercise: Determination of percentage of iron from the given iron ore sample

1. Aim and Objectives

Aim: To determine the percentage of iron in the given hematite ore

Objective: Determination of percentage of iron in the given hematite ore by external indicator method

2. Apparatus: Burette, pipette, conical flask, water bath, funnel, wax paper etc

3. Introduction:

Haematite is an important ore of iron containing mainly Fe_2O_3 and silica. Estimation of iron involves the dissolution of the ore in hydrochloric acid, reducing the ferric (Fe^{3+}) ions in the solution to ferrous (Fe^{2+}) ions using a reducing agent such as stannous chloride and the estimation of ferrous ions so obtained by titrating against an oxidizing agent like potassium dichromate.

Significance of Iron Estimation: It is very important to assess the quality of iron ore before the price is fixed per ton of the ore material. Also one can decide on the purification method if the concentration of iron is known in any waste stream.
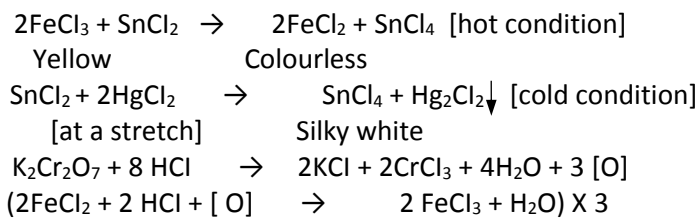
4. Experimental Procedure:

Part - A: Preparation of potassium dichromate solution:

1. Weigh accurately the given potassium dichromate crystals and transfer onto the funnel placed on a 250 cm^3 volumetric flask. Dissolve by adding deionized water and make it upto the mark.
2. Shake well to get uniform concentration.

Part - B: Estimation of Iron:

1. Pipette out 25 cm^3 of the given haematite solution into a clean conical flask. Add 1/4 test tube of concentrated hydrochloric acid.
2. Heat the solution nearly to boiling. Add stannous chloride drop by drop to the hot solution until the solution becomes colorless.
3. Add 2-3 drops of stannous chloride in excess. Add 2 test tubes of deionized water and cool the solution to room temperature, followed by addition of 3 cm^3 of mercuric chloride at a stretch.
4. A silky white precipitate is formed. Reject the contents of the flask and repeat the experiment if no precipitate or greyish precipitate is formed.
5. Titrate the solution against standard potassium dichromate taken in the burette, using potassium ferricyanide as an external indicator.
6. In the beginning, take out a drop of the reaction mixture using a clean glass rod and mix it with a drop of the indicator arranged on paraffined paper.
7. The colour of the drop of indicator changes to blue.
8. Take out a drop of the reaction mixture after every addition of $K_2Cr_2O_7$ and mix it with a fresh drop of the indicator. Appearance of blue or green colour indicates that the end point is not reached.
9. At the end point, a drop of the reaction mixture fails to give either blue or green coloration.

Note down the burette reading and repeat the experiment for agreeing values.

Chemical Reaction:

$$2FeCl_3 + SnCl_2 \rightarrow 2FeCl_2 + SnCl_4 \text{ [hot condition]}$$

Yellow   Colourless

$$SnCl_2 + 2HgCl_2 \rightarrow SnCl_4 + Hg_2Cl_2\downarrow \text{ [cold condition]}$$

[at a stretch]   Silky white

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

$$(2FeCl_2 + 2\ HCl + [\ O] \rightarrow 2\ FeCl_3 + H_2O)\ X\ 3$$

$$K_2Cr_2O_7 + 14\ HCl + 6FeCl_2 \rightarrow 2KCl + 6FeCl_3 + 2CrCl_3\ (Green)\ + 7H_2O$$

On the paraffined paper, before the end point: $3Fe^{2+} + 2\ [Fe\ (CN)_6]^{3-} \rightarrow Fe_3[Fe(CN)_6]_2$

(bright yellow)   blue

5. **Data Collection and Tabulation:**

Standard Potassium Dichromate Solution in the burette	Trial 1	Trial 2	Trial 3
Final burette reading (ml)			
Initial burette reading (ml)			
Volume of potassium dichromate consumed (ml)			

6.   **Calculation:**

Part-A : Preparation of potassium dichromate solution:

Weight of $K_2Cr_2O_7$ + weighing bottle, w_1 = ----------------g

Weight of empty weighing bottle, w_2 = ------------------g

Weight of the $K_2Cr_2O_7$ transferred, w = -------------------g

Normality of $K_2Cr_2O_7$ = $\dfrac{\text{Weight of } K_2Cr_2O_7 \text{ crystals dissolved in 250 cm}^3\ X\ 4}{\text{Gram equivalent weight of } K_2Cr_2O_7\ (49)}$ =   xN

Volume of potassium dichromate consumed (consider concordant burette reading or least of the three readings)   = Y   ml

1000 cm^3 of 1N $K_2Cr_2O_7$ = 1 equivalent of iron= 55.85 g of Iron

Therefore 'Y' cm^3 of 'x' N $K_2Cr_2O_7$ =$\frac{55.85 \, x \, X \, x \, Y}{1000}$ g of iron =   = 'Z'   g of iron.

25cm^3 of haematite ore solution contains 'Z'   g of iron

250 cm^3 of haematite ore solution contains '10 Z' g of iron =

Weight of haematite ore dissolved in 250 cm^3 of the solution = 'W' g =

'W' g of haematite ore contains '10 Z' g of iron.

Therefore, Percentage of haematite ore = $\frac{10Z \, x \, 100}{W}$ =

Result: Percentage of iron in a given sample of haematite ore   =

7.   **Comments:**

a. Limitations of Experiments

b. Limitations of Results

c. Learning happened

d. Recommendations

Assessment:

Short Procedure	(2.5 marks)	
Performance/ Observation	(12 marks)	
Calculation + Graph	(3 marks)	
Viva-voce	(7.5 marks)	
Total	(25 marks)	

Title of the Laboratory Exercise: Determination of efficiency of electroplating of copper

1. Aim and Objectives

Aim: To determine the efficiency of electroplating of copper

Objective: Determination of percentage efficiency of copper using copper as an anode on an iron cathode

2. Apparatus: Apparatus: Periodic Table, Calculator, Glass beaker, Iron nail,2 wires (use as electrodes),Kimwipes, Lab timer, Amp meter, Laptop PC with *Copper Plating Efficiency* Excel spreadsheet, Small pieces of sandpaper (100 grit)

3. Introduction:

The purpose of this experiment is to demonstrate some principles of chemistry through the use of electroplating copper (Cu) from solution. Electroplating can be used to deposit a layer of metal such as chromium, copper, gold, nickel, or zinc on another metal. This deposit can provide a protective and decorative coating for the metal which lies beneath it.

Significance: Electroplating is very important in various industrial applications. Electroplating of copper is done to obtain high electrical conductivity, decorative purposes, etc.

Components of an Electroplating:

Electroplating bath:
- Contains a suitable salt solution of the metal being plated ($CuSO_4$)
- Additives (Buffer – Rochelle Salt + NaOH, Complexing agent – Na_2EDTA)

Anode:
- May be a rod or pellets of the metal being plated (Cu) or may be an inert electrode (Pt)
- Should be electrically conducting

Cathode:
- Article to be plated (Iron/Zn)
- Should have an electrically conducting surface

Electroplating of a metal (Me) with copper in a copper sulfate bath
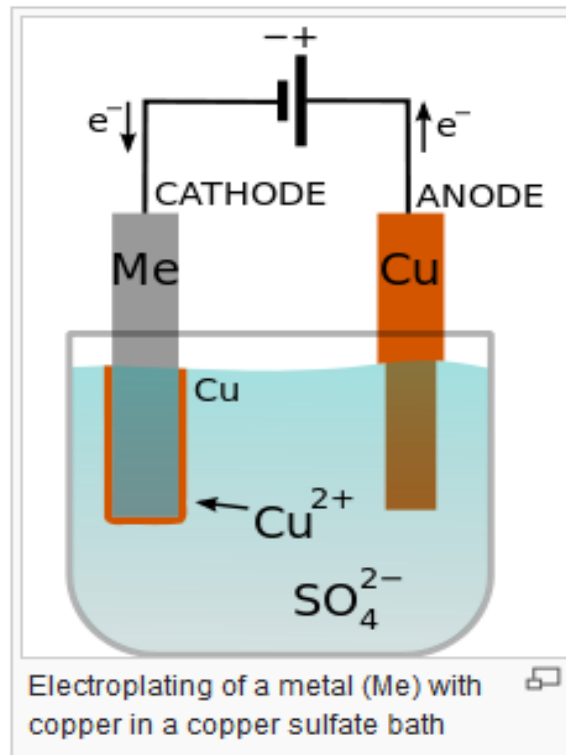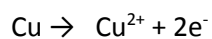
Inert vessel:

- Contains above mentioned materials

- Vessel may be made of rubber lined steel, plastic, concrete or wood
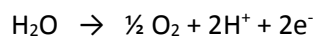
DC power supply:

- Positive terminal of the power supply is connected to the anode

- Negative terminal is connected to the cathode

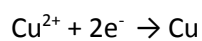**Reactions at anode and cathode during electroplating**:
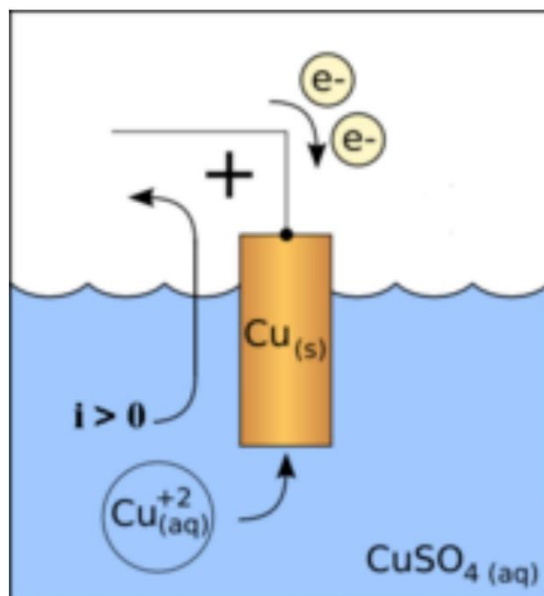
- Oxidation takes place at anode

  $Cu \rightarrow Cu^{2+} + 2e^-$

- When the anode used is an insoluble (inert) anode, oxygen evolution occurs at the anode:

  $H_2O \rightarrow \frac{1}{2} O_2 + 2H^+ + 2e^-$

- Reduction occurs at cathode

- Metal gets deposited on the cathode surface

  $Cu^{2+} + 2e^- \rightarrow Cu$

4. **Experimental Procedure**
    1. Weigh the cleaned (alkali wash, solvent wash, acid wash followed by water wash, then drying) iron nail
    2. Connect the iron nail to the wire clip provided and Connect this wire to the negative terminal of the power supply
    3. Connect the copper rod to the wire clip provided and Connect this wire to the positive (+) terminal of the power supply
    4. Set the voltage at 3 volts
    5. Carry out electroplating for 20 minutes
    6. At the end of 20 minutes, take out the nail, dry it and record the weight.

5. **Calculations:**

    - Weight of nail after plating (g): _____ (g)

    - Weight of nail before plating: _____ (g)

    - Weight of copper deposited = Weight of nail after copper plating – weight of nail before
      Plating: _____ (g)

    - Timer reading for copper plating : _____ min and _____ sec

    - Current Reading _____ (amps)

      Atomic weight of copper = 63.54 g mol

      Number of moles (Φ) plated = $\dfrac{\text{Weight of copper plated on nail}}{\text{Atomic weight of copper}}$

      Number of moles (Φ) of copper plated: _____ (moles)

      Efficiency %   = $(W_1 / W_2) * 100$
      Where (W_1) is the experimental weight of the deposit and (W_2) is the theoretical weight of the deposit (W_2 will be provided).

6. **Result:** …….. % Efficiency

7. **Conclusions**

8. **Comments**

   a. Limitations of Experiments

   b. Limitations of Results

   c. Learning happened

   d. Recommendations

Assessment:

Short Procedure	(2.5 marks)	
Performance/ Observation	(12 marks)	
Calculation + Graph	(3 marks)	
Viva-voce	(7.5 marks)	
Total	(25 marks)	

Title of the Laboratory Exercise: Anodizing of aluminium and measurement of its thickness

1. **Aim and Objectives**

    Aim: To carryout anodizing on Aluminium and estimate the thickness layer formed

    Objectives: To find the thickness of the anodized aluminium after carrying out anodizing

2. **Apparatus:** Aluminium wire, beaker, DC source, Inert electrode and sulphuric acid

3. **Introduction:**

Anodizing is a process for producing decorative and protective films on articles made from aluminium and its alloys. It is essentially a process where a thick film of aluminium oxide is built up on the surface of the aluminium through the use of a direct current electrical supply. In the majority of anodizing plants in New Zealand it is carried out in an electrolyte bath containing sulphuric acid with aluminium sheet cathodes and the work to be anodized attached to the anode (**Figure 1**).
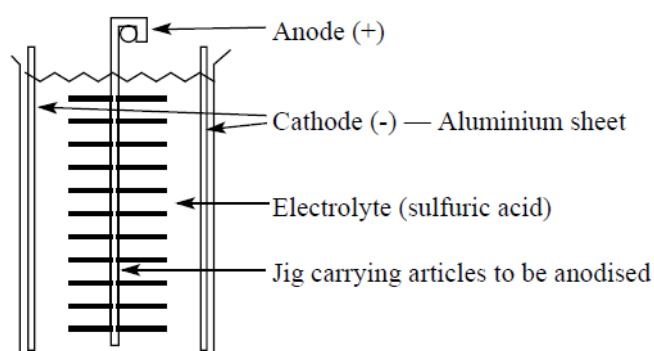
Figure 1 - A typical anodising cell
(Articles have to be securely fastened to ensure electrical contact during the anodising)
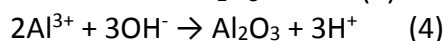
When the current is flowing in the cell the following sequence of events is believed to occur. Sulphuric acid begins to decompose, the hydrogen ions moving to the cathode where they are reduced to hydrogen gas:
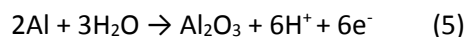
$$2H^+ + 2e- \rightarrow H_2(g) \qquad (1)$$
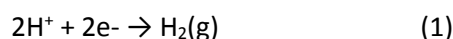
Simultaneously, negatively charged anions, i.e. hydroxide, sulphate and maybe oxide ions move to the anode. The electrical charge in the circuit causes positively charged aluminium ions (Al^{3+}) to be generated in the anode and in turn move toward the cathode. At the anode surface they react with the oxide/hydroxide ions to form aluminium oxide (in the case of the hydroxide ion, hydrogen ions are released into the solution).

Equations of the anode reactions:

$$Al \rightarrow Al^{3+} + 3e^- \qquad (2)$$

$$2Al^{3+} + 3O^{2-} \rightarrow Al_2O_3 \qquad (3)$$
$$2Al^{3+} + 3OH^- \rightarrow Al_2O_3 + 3H^+ \qquad (4)$$

For which the overall process is:

$$2Al + 3H_2O \rightarrow Al_2O_3 + 6H^+ + 6e^- \qquad (5)$$

The sulphate ions also play some part as the oxide coating contains 12 - 15% sulphate ions. It is suggested that the sulphate ions facilitate the movement of hydrogen ions reducing the cell voltages required.

4. **Experimental Procedure**

   1. Take 30 ml of 50% sulphuric acid in a beaker.

   2. Clean the aluminium wire and weigh it.

   3. Dip the weighed aluminium wire in the beaker containing sulphuric acid and connect it to the anode of the DC source.

   4. Connect the inert electrode to the cathode of the DC source.

   5. Apply 18 Volts of voltage at room temperature for 30 minutes.

   6. Remove the aluminium wire and note down the weight.

5. **Calculations:**

   - Weight of aluminum weir (g): _____ (g)

   - Timer reading for Anodizing of aluminium : _____ min and _____ sec

   - Current Reading _____ (amps)

   - Weight of Anodized aluminium: _____ (g)

Result: The thickness of anodized aluminium is …………

6. **Conclusions**
7. **Comments**

   a. Limitations of Experiments

   b. Limitations of Results

   c. Learning happened

   d. Recommendations

Assessment:

Short Procedure	(2.5 marks)	
Performance/ Observation	(12 marks)	
Calculation + Graph	(3 marks)	
Viva-voce	(7.5 marks)	
Total	(25 marks)	

Title of the Laboratory Exercise: Colorimetric estimation of copper and zinc present in a given brass sample

1. **Aim and Objectives**

    Aim: To estimate the composition of brass

    Objectives: To find the composition of brass alloy using colorimeter

2. **Apparatus:** Pipette, burette, calorimeter, standard flasks, beaker, etc.

3. **Introduction:**
    Colorimetric analysis depends upon the measurement of quantity of light absorbed by a coloured solution. Quantitative measurement of absorption is based on Beer-Lambert's law, which states that, the amount of light absorbed by coloured solution is proportional to the thickness and concentration of the light absorbing medium. It can be expressed as

    Absorbance (or optical density, O.D.), $\log\left(\dfrac{I_0}{I_t}\right) = \varepsilon c t$

    where, 'I_0' is intensity of incident light, 'I_t' is intensity of transmitted light, 't' is thickness of the light absorbing medium (cm), 'c' is concentration of the coloured constituent in the solution (mol/L) and 'ε' is molar extinction coefficient (amount of light absorbed when 't' and 'c' are 1 cm and 1 mol/L respectively). Since, ε is a constant, absorbance is proportional to the concentration of coloured constituent in solution, provided the thickness of the light absorbing medium is kept constant.

    Significance of Colorimetry: The data obtained from colorimeter will be helpful to adjust the color components in food industry and also to monitor the growth of a bacterial or yeast culture. In laboratories, it is used to test water quality for ions such as Cu^{2+}, iron, molybdenum, etc. and also to determine the concentrations of plant nutrients.

    Principle: Diethyldithiocarbamate (DDTC), the common reagent used for spectrophotometric determination, forms $M(DDTC)_2$ complexes with metals such as Cu, Zn, Hg that are soluble in organic solvents like $CHCl_3$ or CCl_4. Though DDTC complex of Cu(II) shows maximum absorption at 435 nm to that of Zn(II). Stability of DDTC complexes with Cu is greater than Zn. Hence from $Zn(DDTC)_2$ zinc is substituted with the addition of excess copper. The subsequent increase or decrease in absorption is equivalent to the amount of zinc. Therefore, based on this substitution, spectrophotometric method has been proposed for the determination of Zn(II), by the measurement of $Cu(DDTC)_2$.

4. **Experimental procedure:**
    1. A known weight of brass is dissolved in 1:1 nitric acid, the obtained solution is treated with small quantity of urea and heated. The solution is further cooled and added with

ammonia till a slight blue precipitate is obtained. This precipitate is treated with 1 ml of acetic acid.

2. 2ml, 4ml, 6ml and 8 ml of this solution is placed in different 50 ml separating flasks containing 0.3 mL 0.05 M H_2SO_4 and 5 mL of the reagent DDTC (5.84×10^{-3} M) solution.

3. Its volume was made up to 15 mL with deionized water. The mixture was stirred for 10 min. The solid product of M(DDTC)$_2$ (Cu and Zn) complexes so formed was extracted carefully with the addition of 10 mL (5×2) $CHCl_3$, and organic phase was separated out. The pH value of one portion was adjusted to 5 using acetate buffer.

4. A portion of 5 mL extract was separated out and its absorbance, which is equivalent to copper, was measured at 435 nm against a blank.

5. Excess Cu(II) solution of 2×10^{-4} M was added to remaining portion (10 mL) of extract when additional Cu(DDTC)2 was formed by the replacement of zinc immediately. Organic phase (10 mL) was further separated after vigorous stirring for 10 min.

6. Another portion of 5 mL extract was separated out and the absorbance was measured at 435 nm against a blank. Additional absorbance is equivalent to Zn(II) present in sample.

7. Then known amount of Hg(II) solution was added to third portion (5 mL) of the extract when it's pH value was adjusted to 10 using ammonium (basic) buffer.

8. The intensity of yellow colour of Cu(DDTC)$_2$ was reduced immediately and organic phase was further separated after vigorous stirring for 10 min.

9. The absorbance was measured at 435 nm against a blank. Reduced absorbance will be equivalent to mercury present in solution.

5. Calculations:

Volume of copper and zinc sulphate (ml)	Absorbance
5	
10	
15	
20	
25	
Test solution(given)	

Volume of copper sulphate (ml)	Absorbance
5	
10	
15	
20	
25	
Test solution(given)	

To calculate copper and zinc:

$1cm^3$ of the stock solution contains =------a-----mg of $CuSO_4.ZnSO4.5H_2O$

{1 molecule of $CuSO_4.ZnSO4.5H_2O \equiv$ 1 atom of Cu and Zn)

499.23 mg of $CuSO_4.ZnSO4.5H_2O \equiv$ 128.93mg of Cu and Zn

a-------- mg of $CuSO_4.ZnSO4.5H_2O \equiv$ $\underline{128.93 \times \text{- - - - - - -}}$ =   mg of Cu and Zn
                                                 499.23

$1cm^3$ of the stock solution contains = b -----------mg of Cu and Zn

v----- ------cm^3 of the test solution contains = b ------- x v ---- mg of Cu and Zn

                                        = ...x...   mg of Cu and Zn

To calculate copper:

$1cm^3$ of the stock solution contains =------a-----mg of $CuSO_4.ZnSO4.5H_2O$

{1 molecule of $CuSO_4.5H_2O \equiv$ 1 atom of Cu

249.54mg of $CuSO_4.ZnSO4.5H_2O \equiv$ 63.54mg of Cu

a-------- mg of $CuSO_4.5H_2O$ $\equiv \dfrac{63.54 \times \text{- - - - -}}{249.54}$ mg of Cu}

$1cm^3$ of the stock solution contains = b -----------mg of Cu

v----- ------cm^3 of the test solution contains = b ------- x v ---- mg of Cu

=...y...mg of Cu

Quantity of Zinc   = x mg of Cu and Zn – y mg of Cu

                      =   =   mg of Zn

Result:
1. The quantity of Cu and Zn in the test solution is ……………mg
2. The quantity of Cu in the test solution is ……………mg
3. The quantity of Zn in the test solution is ……………mg

6. Conclusions

7. Comments

a. Limitations of Experiments

b. Limitations of Results

c. Learning happened

d. Recommendations

Assessment:

Short Procedure	(2.5 marks)	
Performance/ Observation	(12 marks)	
Calculation + Graph	(3 marks)	
Viva-voce	(7.5 marks)	
Total	(25 marks)	

Title of the Laboratory Exercise: Measurement of voltage in a hydrogen-oxygen fuel cell

1. **Aim and Objectives**

   Aim: To determine the voltage generated in a hydrogen – oxygen fuel cell
   Objectives: Determination of voltage in a hydrogen – oxygen fuel using voltmeter

2. **Apparatus:** Hydrogen – oxygen fuel cell, voltmeter, hydrogen gas, etc.

3. **Introduction:**

   Anode (-) and Cathode (+) on each side of the fuel cell are separated by an electrolyte (KOH or NaOH). Hydrogen gas is channeled through anode side and Oxygen through cathode. Platinum catalyst oxidizes hydrogen atoms into H^+ ions and releases electrons. Electrons pass along external circuit; conduct electricity before entering cathode. Electrolyte allows H^+ to pass into the cathode. In cathode, catalyst combines H^+, O^{2-} and electrons, forming H_2O and heat.
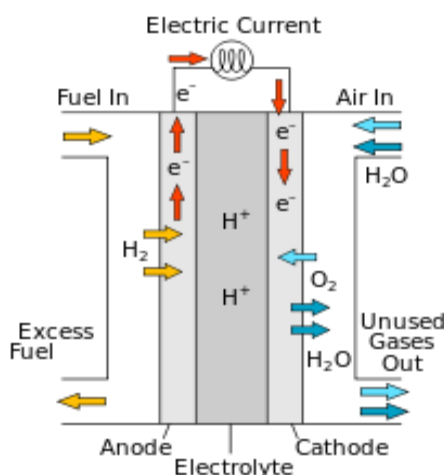
   Fuel cell consists of two electrodes made of porous graphite impregnated with a catalyst such as Pt or Ni or Cd at Anode and CoO at cathode. Electrodes are separated by polystyrene sulphonic acid ion exchange membrane in KOH or NaOH (Electrolyte). In a typical fuel cell, gas (hydrogen-fuel) is fed continuously to the anode (negative electrode) compartment and an oxidant (e.g. oxygen from the air) is fed continuously to the cathode (positive electrode) compartment. Electrochemical reactions take place at the electrodes to produce electric current. Electrodes catalyze the electrochemical reactions and electrolyte facilitates the transfer of hydrogen ions from the anode to the cathode. Membrane allows the passage of protons, but not electrons.

   Hydrogen enters the fuel cell at the anode (negative electrode) where it is **oxidized** into two H+ ions (protons) and 2 electrons for every hydrogen molecule. Following are the **electrochemical reactions**.

   $$2H_2 \rightarrow 4H^+ + 4e-$$
   $$O_2 + 4H^+ + 4e^- \rightarrow 2 H_2O$$

$$2H_2 + O_2 \rightarrow 2H_2O$$

Fuel cell: Hydrogen and oxygen gases flow into the cell producing water and electricity.

Electrolysis cell: Hydrogen and oxygen gases produced when electricity flows through an electrolyte containing water.
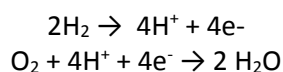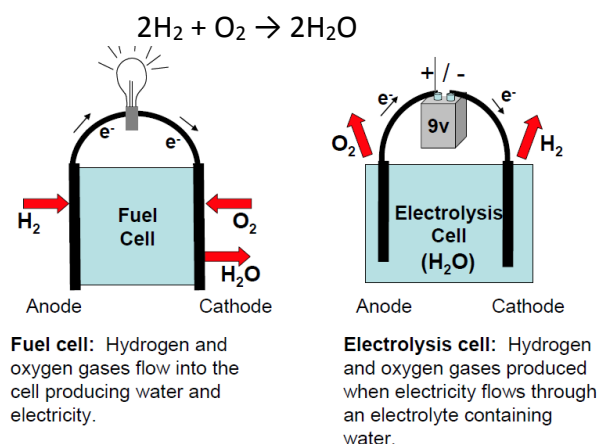
$$\% \text{ efficiency} = 100\% \times \frac{\textit{Voltage out}}{\textit{Voltage max}} \%$$

Significance: Hydrogen – oxygen fuel cells are gaining importance as they are eco-friendly. They do not produce any harmful gases. These fuel cells are used to power automobiles, in military applications, etc.

4. **Experimental Procedure**:
    1. Hydrogen gas is fed continuously to the anode compartment
    2. Oxygen is drawn from air (which is used as an oxidant) continuously to the cathode compartment
    3. Voltage generated is recorded using voltmeter

    Result: Voltage generated =   V

5. **Conclusions**

6. **Comments**

    a. Limitations of Experiments

    b. Limitations of Results

    c. Learning happened

    d. Recommendations

Title of the Laboratory Exercise: Determination of calorific value of a fuel sample using Bomb calorimeter

1. **Aim and Objectives**

   Aim: To determine the calorific value of a fuel
   Objectives: Determination of calorific value of a fuel using Bomb Calorimeter

2. **Apparatus:** Bomb Calorimeter, fuel sample, DC power source, oxygen supply, etc.

3. **Introduction:** Calorific value of the fuel is the quantity of heat liberated when unit quantity of fuel is burned completely. When hydrogen is present in the fuel, it is converted to steam. Thus, higher heating value (also called gross calorific value) is the total amount of heat liberated when one unit of fuel is burnt completely and the combustion products are brought to their room temperature. Lower heating value (also called net calorific value) is Net calorific value = Gross calorific value – latent heat of condensation.

   Heat liberated during the combustion of known weight of fuel is equal to the heat absorbed by water and copper calorimeter. Gross calorific value of the fuel is determined by measuring the temperatures before (T_1) and after (T_2) combustion of the fuel.
   The parts of a bomb calorimeter are shown in figure given below:

   Bomb calorimeter
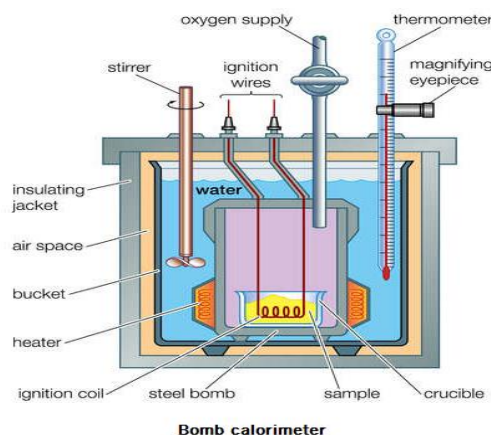
   Significance: Calorific value of a fuel depends on the carbon and hydrogen content. It is important to determine the calorific value of a fuel sample, so that the quality of fuel can be assessed. Higher calorific value containing fuel is a good fuel.

4. **Experimental Procedure**:
   1. Weigh about 1 g of sample and introduce it into the bomb.
   2. Connect the fuse to the lid wires.
   3. Pressurize the bomb with oxygen and place it inside the jacket.
   4. Fill the jacket with 2000ml of water. Temperature of water must be 2°C less than the room temperature.

5. Assemble the calorimeter and run the stirrer for 5 minutes.
6. Record the temperature of the water bath accurately using a precision thermometer at an interval of 1 minute for 5 minutes and ignite the charge at the start of 6th minute.
7. Keep recording the temperature every minute till constant temperature is attained.
8. Open the bomb carefully.
9. Clean the apparatus after it attains room temperature.
10. Collect and weigh the residual ash.

Caution: Never approach the top of the bomb due to danger of explosion

5. Observation and calculations:

- Initial temperature, t_1 =   °C

- Final temperature, t_2 =   °C

- Rise in temperature $\Delta t = t_2 - t_1$ =   °C

- Weight of fuel sample, m =   kg

- Weight of water taken in the calorimeter, w_1 =   kg

- Weight of water equivalent of calorimeter, w_2 =   kg

- Specific heat of water, s =   J kg^{-1} °C^{-1}

GCV, $Q = \dfrac{(w_1 + w_2) \times s \times \Delta t}{m}$

    =

Title of the Laboratory Exercise: Demonstration of producing printed circuit board

1. Aim and Objectives

Aim: To prepare a printed circuit of Cu on a plastic board
Objectives: Preparation of printed circuit board on a plastic board using electroless plating method.

2. Apparatus: $CuSO_4$, Na_2EDTA, Buffer solution (Rochelle salt + NaOH), $FeCl_3$, HCl, Plastic board, etc.

3. Introduction:

A printed circuit board, or PCB, is a self-contained module of interconnected electronic components found in devices ranging from common beepers, or pagers, and radios to sophisticated radar and computer systems. The circuits are formed by a thin layer of conducting material deposited, or "printed," on the surface of an insulating board known as the substrate. Individual electronic components are placed on the surface of the substrate and soldered to the interconnecting circuits. Contact fingers along one or more edges of the substrate act as connectors to other PCBs or to external electrical devices such as on-off switches. A printed circuit board may have circuits that perform a single function, such as a signal amplifier, or multiple functions.

There are three major types of printed circuit board construction: single-sided, double-sided, and multi-layered. Single-sided boards have the components on one side of the substrate. When the number of components becomes too much for a single-sided board, a double-sided board may be used. Electrical connections between the circuits on each side are made by drilling holes through the substrate in appropriate locations and plating the inside of the holes with a conducting material. The third type, a multi-layered board, has a substrate made up of layers of printed circuits separated by layers of insulation. The components on the surface connect through plated holes drilled down to the appropriate circuit layer. This greatly simplifies the circuit pattern.

Significance: A printed circuit board are used in pagers, and radios to sophisticated radar and computer systems

PCB starts as a fiberglass sheet about 16" x 20" x .062"

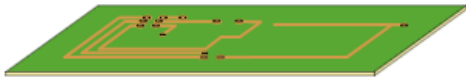Thin copper sheets are added to both sides

Etchant-resistive wire pattern printed on one or both sides

All copper removed with etchant except for wires and pads

Holes drilled for leads and vias, and plated with metal

Soldermask applied to keep solder only where needed

4. **Procedure:**

1. Two sides of a plastic board is electro-less plated with copper by dipping into the electroless plating bath for 30 minutes, followed by drying.

2. Circuit is printed on the board using an enamel and dried.

3. Board is dipped in acidified ferric chloride which etches away copper leaving the circuit drawn portion.

4. Enamel is removed using an organic solvent to get the required circuit on board.

Title of the Laboratory Exercise: Preparation of a polymer composite by hand layup technique

1. **Aim and Objectives**

   Aim: To prepare a polymer composite
   Objectives: Preparation of a polymer composite by hand layup technique.

2. **Apparatus:** Epoxy or unsaturated polyester resin, hardener, accelerator, stirrer, mold, etc.

3. **Introduction:** Polymer composites are integral components of everyday life. They can be found in aerospace, automobile, structural and in domestic applications. Since the weight to strength ratio is very high for these composites, they have become very popular. Hand layup technique is very simple technique in producing composite materials. It is carried out by laying the resin layer and matrices layer alternatively and compressing between two hot plates to cure the material.

   Significance: The tremendous growth of polymer composite materials industry is because of their aspect ratio. One can find the composite materials in almost all applications. Depending on the requirement the resin, filler system and the technique of preparation can be chosen.

4. **Procedure:**

   1. A known quantity of polymeric resin is taken into a beaker.
   2. 85% by weight of hardener and 2% by weight of accelerator is added into epoxy and mixed thoroughly by stirring at a speed of 1200 rpm in a mechanical stirrer for 10 minutes. A portion of this mixture can be layered onto to a steel plate.
   3. A layer of filler (ex: glass fiber matrices or any other compatible fabric matrices) can be layered next onto resin layer.
   4. Resin and filler layer can be layered alternatively to the required thickness.
      [Alternatively 1% by weight of a filler (clay or wood powder or short fiber) can mixed with epoxy resin and transferred into a metal mold. Then it can be cured at oven at 80°C for 2 hours and post-cured at 100°C for next 4 hours]
   5. The mold is cured in an oven at 80°C for 2 hours and then post-cured at 100°C for 4 hours.

   Note: For a new composite material further activities include measurement of mechanical, thermal, morphological and electrical properties to assess the suitability of the material for a specific application.

Title of the Laboratory Exercise: Construction and Working of Lead Acid Battery

1. Aim and Objectives

Aim: To construct a lead acid battery and measure voltage produced

Objectives: Construction and Working of Lead Acid Battery using lead and lead oxide electrodes with sulphuric acid as electrolyte

2. Apparatus: Lead, Lead oxide electrodes, sulphuric acid, plastic container, volt meter. etc.

3. Introduction:

The battery is an essential component of almost all aircraft electrical systems. Batteries are used to start engines and auxiliary power units, to provide emergency backup power for essential avionics equipment, to assure no-break power for navigation units and fly-by-wire computers, and to provide ground power capability for maintenance and pre-flight checkouts. Many of these functions are mission critical, so the performance and reliability of an aircraft battery is of considerable importance. Other important requirements include environmental ruggedness, a wide operating temperature range, ease of maintenance, rapid recharge capability and tolerance to abuse.

Batteries operate by converting chemical energy into electrical energy through electrochemical discharge reactions. Batteries are composed of one or more cells, each containing a positive electrode, negative electrode, separator, and electrolyte. Cells can be divided into two major classes: primary and secondary. Primary cells are not rechargeable and must be replaced once the reactants are depleted. Secondary cells are rechargeable and require a DC charging source to restore reactants to their fully charged state. Examples of primary cells include carbon-zinc (Leclanche or dry cell), alkaline-manganese, mercuryzinc, silver-zinc, and lithium cells (e.g., lithium-manganese dioxide, lithium-sulfur dioxide, and lithiumthionyl chloride). Examples of secondary cells include lead-lead dioxide (lead-acid), nickel-cadmium, nickel-iron, nickel-hydrogen, nickel-metal hydride, silver-zinc, silver-cadmium, and lithium-ion. For aircraft applications, secondary cells are the most prominent, but primary cells are sometimes used for powering critical avionics equipment

Significance: Secondary batteries like lead acid battery used as storage devices and used to start engines and auxiliary power units, to provide emergency backup power for essential avionics equipment.
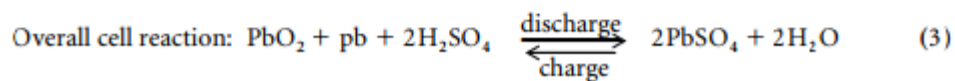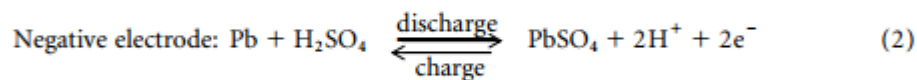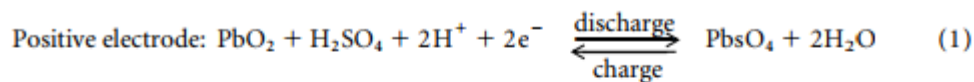
Construction:

- Anode and Cathode are made up of lead grids
- Anode grid is packed with spongy lead
- Cathode grid is packed with PbO_2
- Electrode pairs are separated by a separator made up of polyethylene
- Electrodes are dipped in an electrolyte of about 37 % H_2SO_4 with specific gravity 1.25

- In the discharged state both electrodes turn into lead sulfate and the electrolyte is consumed during the process
- Reverse reactions occur during recharge
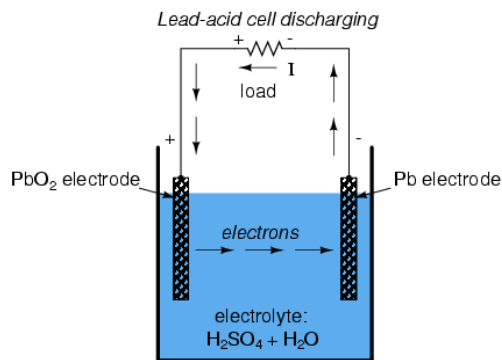
Working of Lead Acid Battery

The chemical reactions that occur in a lead-acid battery are represented by the following equations:

$$\text{Positive electrode: } PbO_2 + H_2SO_4 + 2H^+ + 2e^- \underset{\text{charge}}{\overset{\text{discharge}}{\rightleftarrows}} PbsO_4 + 2H_2O \qquad (1)$$

$$\text{Negative electrode: } Pb + H_2SO_4 \underset{\text{charge}}{\overset{\text{discharge}}{\rightleftarrows}} PbSO_4 + 2H^+ + 2e^- \qquad (2)$$

$$\text{Overall cell reaction: } PbO_2 + pb + 2H_2SO_4 \underset{\text{charge}}{\overset{\text{discharge}}{\rightleftarrows}} 2PbSO_4 + 2H_2O \qquad (3)$$

- During discharging reactions the specific gravity of sulphuric acid comes down

- When it reaches 1.2 the battery needs to be recharged

- Voltage output is 2V

- During recharging the above cell reaction is reversed and sulphuric acid is regenerated

Title of the Laboratory Exercise: Determination of empirical formula of a fuel using exhaust gas analyser

1. **Aim and Objectives**

Aim: To determine the empirical formula of a fuel
Objectives: Determination of empirical formula of a fuel using Orsat analyser

2. **Apparatus:** Orsat analyser and Fuel

3. **Introduction:**

Flue gases are the outlet gases obtained when a fuel is burned with air in combustion equipments like engines and boilers. Based on the fuel composition flue gases are formed, a fuel having carbon and hydrogen compounds generates flue gas containing oxides of carbon and hydrogen because the oxygen in air burns with components that release heat energy and resulting the combusted materials. In construction of fire heater, boilers, combustion engines and combustor the outlet is given out with a cylinder shape chimney so that the hot flue gases are ejected into the atmosphere. In design and calculating the performance of the above equipments the flue gas is used for analysis which help to determine the composition of the carbon dioxide, oxygen, nitrogen and carbon monoxide. Based on this analysis results the fuel inlet flow rate, air flow rate and burner are controlled to get the efficiency of the fired heaters. By the following presentation the flue gas analysis can be demonstrated by using Orsat apparatus, of course this is a standard piece of equipment. Many high accurate digital and portable devices are available to determine the composition of flue gas.

Working of Orsat anlalyser:

- Filling the flue gas
- Expelling excess gas
- Absorption of the gas through contact with chemical by rising and lowering the levelling bottle
- Finally measuring the percentage of component by equating the water levels, repeating (3 and 4) with other two absorber chambers.
  To understand Orsat apparatus operation, representation with single absorber chamber is given. This is performed with other chemical absorption units for same inlet gas.

Orsat apparatus operation shown
with single absorber chamber
www.inclusivescienceengineering.com
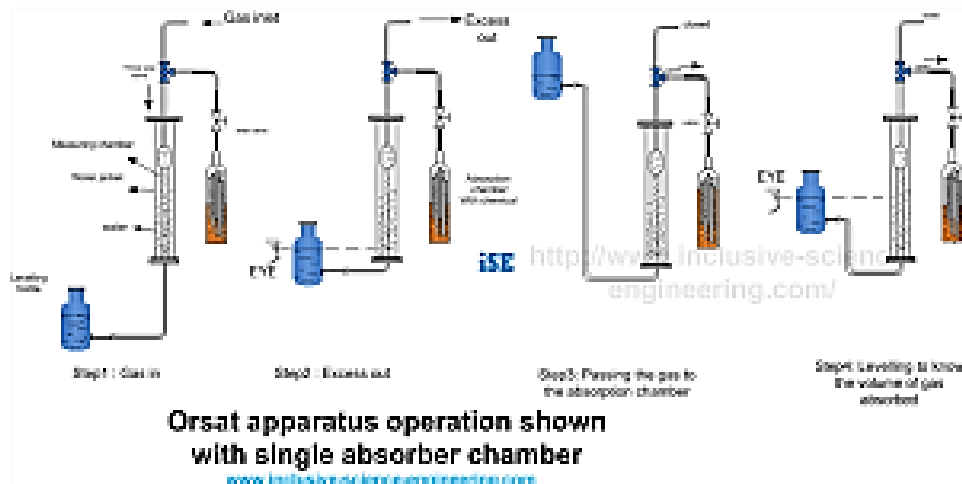
Governing chemical absorption reactions in Orsat apparatus:
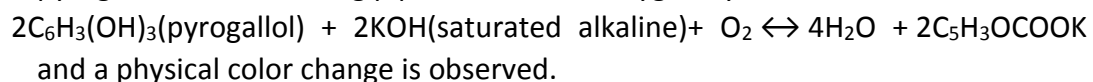
Typical flue gas analyzers measure the quantity of carbon dioxide, carbon monoxide and oxygen by a chemical absorption principle. Based on the absorption factor of these three components their respective absorbing solutions are selected in three different bulb compartments. When the gas is passed into these bulbs consecutively, where each component is separated in sequence that helps to know the volume drop from initial flue gas volume.

4. **Experimental procedure**

1. Water vapor in flue gas is removed by adsorption on solid calcium chloride and then passed into three pipettes.
2. The sequence of absorption should always be started from CO_2, O_2 and CO if the absorption solutions are ammoniacal cuprous chloride; alkaline pyrogalic acid and KOH were selected as reagent in the Orsat apparatus analyser.
3. Gas then is passed into KOH (potassium hydroxide) solution pipette to absorb CO_2 to form potassium carbonate by the reaction

    $2KOH + CO_2 \leftrightarrow K_2CO_3 + H_2O$ at ambient conditions.
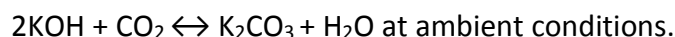
4. After confirming no change in the volume of reservoir, then gas is led to alkaline pyrogallic acid containing pipette to absorb oxygen by the reaction:
   $2C_6H_3(OH)_3(pyrogallol) + 2KOH(saturated\ alkaline) + O_2 \leftrightarrow 4H_2O + 2C_5H_3OCOOK$
   and a physical color change is observed.
5. Finally carbon monoxide is absorbed by ammoniacal cuprous chloride pipette by the reaction,
    $2CuCl + 2CO \rightarrow (in\ NH_3\ solution) \rightarrow [CuCl(CO)]_2$
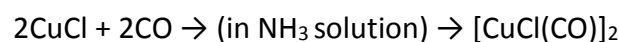
6. Average molecular weight of the flue gas can be calculated using the volume or mole percentage composition determined by the Orsat analysis.

1. Determination of viscosity coefficient of given liquid Using Ostwald's viscometer

- Fix a clean & dry viscometer vertically in a thermostat
- Pipette out known quantity of the given organic liquid into wider limb of the Ostwald's viscometer and raise the liquid into the capillary limb
- Determine the time for its flow from upper mark (above the glass-bulb) to lower mark (below the glass-bulb) for three readings
- Pour out the liquid, wash, rinse with acetone & dry it in the oven and then cool
- Repeat the procedure with same quantity of distilled water to determine the flow time for water
- Knowing the densities of water and test liquid, and viscosity of distilled water at thermostat temperature, the viscosity of the test liquid can be calculated.

  Viscosity coefficient of liquid, $\mu_l = \mu_w \dfrac{t_l\, \rho_l}{t_w\, \rho_w}$

  where, t_l, t_w are flow times and ρ_l, ρ_w are the densities of the test liquid and distilled water respectively

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2. Conductometric estimation of a strong acid using standard NaOH solution

- Dip the conductivity cell in the given acid solution and note down the specific conductance of the solution before the addition of NaOH
- Add standard NaOH solution from the burette in increments of 0.5 mL, stir the solution and note down the specific conductance
- As the titration proceeds, the specific conductance decreases gradually then rises after reaching neutralization point
- Plot a graph of specific conductance against volume of NaOH
- Knowing the normality and quantity of NaOH required for neutralization, quantity of HCl is calculated

Specific Conductance (S m⁻¹) vs Volume of NaOH in cm³ — Equivalence Point

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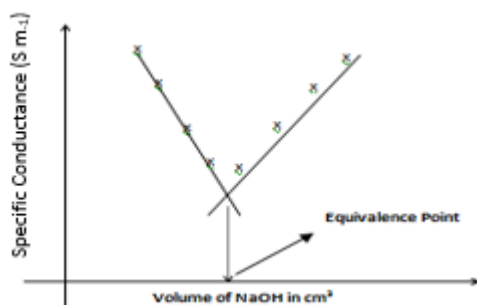
3. Potentiometric estimation of FAS using standard $K_2Cr_2O_7$ solution

- Add half test tube full of dilute H_2SO_4 to the beaker containing FAS solution and immerse the calomel electrode & platinum electrode assembly which are connected to a potentiometer

- Measure the cell potential before the addition of $K_2Cr_2O_7$

- Add $K_2Cr_2O_7$ from a micro-burette in increments of 0.5 ml, mix the solution and note down the cell potential after each addition

- A sudden rise in cell potential is observed at the equivalence point, continue the addition of $K_2Cr_2O_7$ for further 6-8 readings

- Plot a graph of $\Delta E/\Delta V$ against Volume of $K_2Cr_2O_7$ & determine the equivalence point

- From normality and volume of $K_2Cr_2O_7$ consumed, calculate the normality & weight of FAS in the given solution

$\frac{\Delta E}{\Delta V}$ vs Volume of $K_2Cr_2O_7$ — Equivalence point

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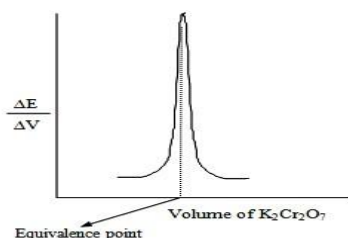
5. Determination of first order rate constant for acid hydrolysis of ethyl acetate

- Take 5 cm³ ethyl acetate and 100 cm³ 0.5N HCl in two different conical flask and allow them to attain room temperature

- Rinse and fill the burette with 0.1N NaOH

- Mix ethyl acetate to 0.5N HCl, shake well

- Pipette out 5 cm³ of above solution and transfer it to conical flask containing ice pieces and phenolphthalein indicator

- Titrate immediately with 0.1N NaOH till pale pink colour persists

- Titrate, **5** cm³ of the same reaction mixture at every **10 minutes** till 30 minutes

- Take 25 cm³ of reaction mixture again and heat at 70°C for 30 min. to accelerate the reaction for completion

- Cool the reaction mixture to room temperature, pipette out 5.0 cm³ into ice cubes and phenolphthalein indicator containing conical flask

- Titrate it as before
- Calculate the 'k' using given formula
- Plot a graph of log (a-x) vs time and calculate k from slope
- Calculate the 'k' using the formula

$$k = \frac{2.303}{t} \log \frac{a}{a-x}$$

Where 'a' is initial concentration and 'a-x' is concentration at time 't'

Time
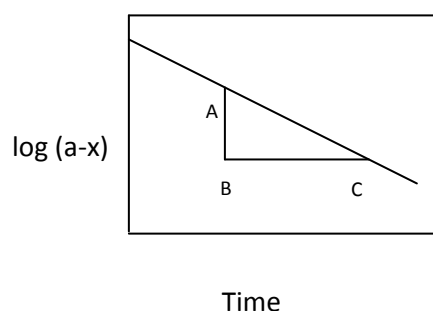
## 6. Determination of percentage of iron from the given iron ore sample

- Weigh accurately the given potassium dichromate crystals and transfer onto the funnel placed on a 250 cm^3 volumetric flask. Dissolve by adding deionised water and make it upto the mark. Shake well to get uniform concentration.
- Burette: Standard $K_2Cr_2O_7$ solution
- Conical flask: 25 cm^3 of the given haematite solution + 1/4 test tube of concentrated hydrochloric acid, heat + Add stannous chloride drop by drop+ . Add 2-3 drops of stannous chloride + Add 2 test tubes of demonized water + 3 cm^3 of mercuric chloride at a stretch
- Indicator: potassium ferricyanide
- End point: colourless to blue color
- From the titre value calculate the percentage of iron in the given sample

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7. Colorimetric estimation of Nickel
Take given quantity of Nickel sulphate in 100ml standard flask to this add 2ml of sulphuric acid + 4 ml of sodium potassium tartrate + 16ml of potassium per sulphate
- To above stock solution add 2 ml of Dimethylglyoxime (DMG) and 5 ml of NaOH keep it aside for an half an hour, after half an hour make it up to mark using distilled water.
- Take 5ml, 10ml, 15ml, 20ml and 25ml of the stock solution into 5 different 50 ml volumetric flasks and a test solution in another volumetric flask.
- Dilute with distilled water up to the mark and shake well.

- Prepare a blank solution by taking all the reagents except nickel sulphate.
- Place the blank solution in the quartz cell (cuvette) of the instrument and set the emission intensity to read zero at 560 nm.
- Measure the absorbance of the standard solutions and test solution with reference to the blank at 560 nm wavelength.
- Plot a graph of absorbance Vs Volume of NiDMG Complex, find the test solution volume and calculate the quantity of Ni present in test solution
- Draw a calibration curve by plotting the absorbance (y-axis) against volume of NiDMG Complex solution (x-axis).
- From the calibration curve, find out the volume of the given test solution and calculate the amount of Nickel.

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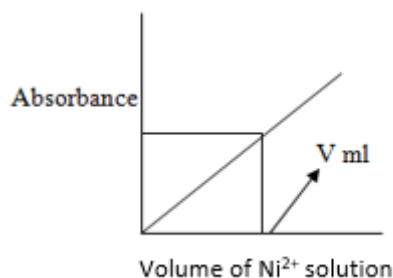
8. **Determination of efficiency of electroplating of copper**
   - Weigh the cleaned iron nail
   - Connect the iron nail to the wire clip provided and Connect this wire to the negative terminal of the power supply
   - Connect the copper rod to the wire clip provided and Connect this wire to the positive (+) terminal of the power supply
   - Time the plating experiment with the lab timer for approximately 10 minutes
   - At the end of a 10 minutes, and record the weight of iron nail which is coated with copper