MANUAL FOR CHEMISTRY PRACTICALS

FOR

ICD CLASSES SECOND SEMESTER

Subject Code: CY-121 (Chemistry-II) CHEMISTRY LAB

DEPARTMENT OF CHEMISTRY SLIET, LONGOWAL (DEEMED UNIVERSITY) DISTT-SANGRUR, PUNJAB-148106

SYLLABUS

L T P Cr 4 0 2 5

LIST OF PRACTICALS

- 1. To determine Iron content in Mohr's salt by standard KMnO₄ solution.
- 2. To study the adsorption of iodine from alcoholic solution by charcoal.
- 3. To study hydrolysis of methyl acetate in presence of hydrochloric acid.
- 4. To detect the extra elements (Nitrogen, Sulphur and Halogens) present in the given organic compound.
- 5. To detect the functional group (any one) Carboxylic acid (-COOH), Phenol, Alcohol (-OH), Aldehyde and Ketone, Ester (-COOR), Acid amide (-CONH₂), amino (-NH₂) present in the given organic compound.

CONTENTS

- 1. To determine iron content in Mohr's salt solution using standard KMnO₄ solution.
- 2. To study the adsorption of Iodine from solution by Charcoal.
- 3. To determine the rate constant for the acid-catalyzed hydrolysis of methyl acetate.
- 4. To detect the extra elements (Nitrogen, Sulphur and Halogens) present in the given organic compound.
- 5. To detect the functional group carboxylic acid (-COOH) present in the given organic compound.
- 6. To detect the functional group phenol present in the given organic compound.
- 7. To detect the functional group Alcohol (-OH) present in the given organic compound.
- 8. To detect the functional group aldehyde and ketone (Carbonyl compounds, >C=O) present in the given organic compound.
- 9. To detect the functional group Ester (-COOR) present in the given organic compound.
- 10. To detect the functional group acid amide $(-CONH_2)$ present in the given organic compound.
- 11. To detect the functional group amines $(-NH_2)$ present in the given organic compound.

Instructions to the Students

- > Keep work area neat and free of any unnecessary objects.
- > Never pour chemical waste into the sink drains or wastebaskets.
- Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
- Wear disposable gloves, as provided in the laboratory, when handling hazardous materials.
- Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
- ➤ Wear shoes that adequately cover the whole foot.
- Keep your hands away from your face, eyes, mouth, and body while using chemicals.
- In case of an emergency or accident, follow the established emergency plan as explained by the teacher and evacuate the building via the nearest exit.
- Always use a spatula to remove a solid reagent from a container.
- Use a hot water bath to heat flammable liquids. Never heat directly with a flame.
- Add concentrated acid to water slowly. Never add water to a concentrated acid.
- > Never place the container directly under your nose and inhale the vapors.
- > Never mix or use chemicals not called for in the laboratory exercise.
- > Clean up all spills properly and promptly as instructed by the teacher.
- Thoroughly clean your laboratory work space at the end of the laboratory session. Do not block the sink drains.

Aim: To determine iron content in Mohr's salt solution using standard KMnO₄ solution.

Requirements:

Apparatus/Glasswares: Burette, pipette, conical flask, burette stand and clamp

Chemicals: KMnO₄, H₂SO₄, Mohr's salt [(NH₄)₂SO₄ FeSO₄.6H₂O].

Theory:

Titrations in which a reducing agent is titrated against an oxidizing agent or vice versa are called Redox titration. In a redox reaction if one substance is oxidized the other is reduced. Oxidation is defined as the process in which loss of electron from the element takes place and reduction is a process where electrons are accepted by the element or the ion. The reagent which undergoes reduction is an oxidizing agent and the reagent which undergoes oxidation is a reducing agent. The oxidation and reduction are complementary to one another and takes place simultaneously.

The equivalent weight of an oxidizing and reducing agent can be obtained by dividing molecular weight of the compound by the total number of electrons that are gained or lost in a reaction per molecule of the substance.

 $MnO_4^- + 8H^+ + 5e \rightarrow Mn^{2+} + 4H_2O$

Therefore, equivalent weight of $KMnO_4 = (Molecular weight of KMnO_4) / 5$

Similarly, for the oxidation of $Fe^{2+} \rightarrow Fe^{3+}$

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

Therefore, Equivalent weight of Ferrous salt = (Molecular weight of Ferrous salt)/1

In this redox titration under experiment $KMnO_4$ oxidizes ferrous ion of the salt to ferric ion in acidic medium and itself reduces manganous (Mn^{2+}) ion.

 $MnO_4^- + 8H^+ + 5e \rightarrow Mn^{2+} + 4H_2O$ [$Fe^{2+} - e = Fe^{3+}$] x 5

 $MnO_4^- + 8H^+ + 5 Fe^{2+} \rightarrow Mn^{2+} + 5 Fe^{3+} + 4H_2O$ (Ionic equation) The actual equation is:

2 KMnO₄ + 10 FeSO₄ + 8 H₂SO₄ \rightarrow K₂SO₄ + 2MnSO₄ + 5 Fe₂(SO₄)₃ + 8 H₂O As the change in color in acid – base indicator depends on the pH of the reaction mixture, the change in color of the indicator in redox titration depends upon the electrode potential of the system. Therefore, a redox indicator has to be chosen in such a way that its potential lies within that of the system. In this titration, KMnO₄, the oxidizing agent is the selfindicator as it is strongly colored (purple) and decolorizes after reduction. Potassium permanganate is purple in its oxidized form (MnO₄⁻) but its reduced form (Mn²⁺) is light pink in acidic medium.

MnO_4^- + $8H^+$ + $5e$ →	$Mn^{2+} + 4H_2O$
(Purple)	(light pink)

As, the titration proceeds, the MnO_4^- will converted to Mn^{2+} and simultaneously Fe^{2+} will converted to Fe^{3+} . When whole of the reducing agent (Fe^{2+}) will consumed, the oxidizing agent (MnO_4^-) cannot be reduced, then the addition even one drop of KMnO₄ solution given its color to the solution. This indicates the end point of the titration.

Procedure:

Estimation of Fe (II) in the given solution: Pipette out 20 mL of unknown Fe(II) solution in a 100 mL conical flask, add 20 mL of dilute H_2SO_4 . Titrate with standard KMnO₄ solution of known strength till the first permanent pink color develops throughout the whole mass of the solution. Take minimum three readings.

Observations:

Table :- Unknown Mohr's salt with KMnO₄ solution.

Sr. N o	Burette R	Volume of KMnO ₄ required	
	Initial	Final	(mL)
1.			
2.			
3.			

$$V_1S_1$$
 = V_2S_2
(KMnO₄) (Mohr's salt)

$$S_2 = \frac{V1S1}{V2}$$

1000 mL of 1(N) KMnO₄ solution = 1 equivalent of Fe = 56 g of Fe

 V_1 mL of $S_1(N)$ KMnO₄ solution = 0.056 x V_1 x S_1 g of Fe

20 mL solution contains (0.056 x 20x S₁) g of Fe 1000 mL solution contains (0.056 x S₁x 50) g of Fe Therefore, Fe present in the solution = (0.056 x V₁x S₁X 50) g/L = A g/L (say) Result can also be expressed in terms of Mohr's salt in g/L. 56 g of Fe present in 392 g of Mohr's salt 'A' g of Fe present in $\frac{392 \times A}{56}$ g of Mohr's salt Therefore, amount of Fe(II) in 'A' g/L and amount of Mohr's salt is $\frac{392 \times A}{56}$ g/L

Discussion:-

- i) Potassium permanganate (KMnO₄) is a strong oxidizing agent in acid medium. Sulphuric acid is the most suitable acid as it has no action upon permanganate in dilute solution.
- ii) With dilute HCl, there is a probability that some amount of permanganate may be consumed in the formation of Cl2 gas and thus interfere the estimation.

 $2MnO_4^- + 10Cl^- + 16H^+ \rightarrow 2Mn^{+2} + 5Cl_2 + 8H_2O$

iii) HNO₃ cannot be used as it itself is a strong oxidizing agent and may oxidize the reducing agent, thereby may cause error in estimation.

Precaution:-

- i) All the glass apparatus should be washed thoroughly with distilled water before use.
- ii) Burette and pipette should be rinsed with the solution to be taken in it.
- iii) There should not be any leakage in the burette.
- iv) KMnO₄ solution should be kept in dark.

v) KMnO₄ solution should not be filtered through filter paper, it should be taken by decantation.

- vi) Freshly prepared KMnO₄ should be used.
- vii) H₂SO₄ should be added in excess otherwise brown precipitate of MnO₂ may appear.

viii) The titration (conical flask) should be placed on white paper or board to identify properly the color change at the end point.

Aim: To study the adsorption of Iodine from solution by Charcoal.

Apparatus: Conical flask, centrifuge tubes, measuring cylinder, analytical balance, centrifuge, burettes, retort stand and clamps, pipette

Materials: Iodine solutions (Table 1), 1% w/v starch solution, 0.1 M sodium thiosulphate solution, distilled water, activated charcoal.

Theory:

Adsorption is a process of free moving of gaseous or solutes molecules of a solution come close and attach themselves onto surface of solid. The adsorption can be strong or weak depends on the nature of forces between solid surface (adsorbent) and the gas or dissolves solute (adsorbate). There are two types of adsorption which is chemical and physical adsorption. Chemical adsorption is when it involves the chemical bond between adsorbent and the adsorbate. It requires activation energy, very strong and not reversible. The physical adsorption is due to the Van der Waals forces between adsorbent and adsorbate. It is reversible, nonspecific and can occur at any condition. Chemical adsorption will produce one layer of the adsorbate while physical adsorption will produce multilayer of the adsorbate. It also can happen where the chemical adsorption is followed by the physical adsorption. The partial pressure of the gas and the concentration of adsorbate will affect the degree of adsorption from the solution. Adsorption isotherm is the relationship between degree of the adsorption and the partial pressure of concentration. The characteristic of the solid form and the reaction involve will be different if there are changes of the isotherm in the temperature. Surface area if the factor that governs the rate of dissolution and the bioavailability of drug that will determine the rate of absorption of the drug thought gastrointestinal tract. The surface area of the solid can be determined by the adsorption measurement. Base on the experiment that has been done, we had study the adsorption of the iodine from the solution and Langmuir isotherm has been used to estimate the surface area of the activated charcoal.

Procedure

		1
Flask	Volume of Solution A (mL)	Volume of Solution B(mL)
1 and 7	10	40
2 and 8	15	35
3 and 9	20	30
4 and 10	25	25
5 and 11	30	20
6 and 12	50	0

Table 1

Solution A: Iodine (0.05M)

Solution B: Potassium iodide (0.1M)

Using burette or measuring cylinders, fill 12 conical flasks (labeled 1-12) with 50 mL mixtures of solution A and B as stated in Table 1.

Set 1: Actual concentration of iodine solution A (X)

For flask 1-6:

- 1) 1-2 drops of starch solution were added as an indicator
- 2) 0.1 M sodium thiosulfate solution was titrated until the colour of the solution changed from dark blue to colourless.
- 3) The volume of the sodium thiosulpahate used was recorded.

Set 2: Concentration of iodine in solution A at equilibrium (C).

For flask 7-12:

- 1) 0.1 g of activated charcoal was added.
- *2)* The flasks were cap tightly. The flask was swirled or shaked every 10 minutes for 2 hours.
- *3)* The solution was transferred into centrifuge tube after 2 hours and was labeled accordingly.
- 4) The solution was centrifuged at 3000 rpm for 5 minutes and the resulting supernatant was transferred into new conical flask. Each conical flask was labeled accordingly.
- *5)* Steps 1, 2 and 3 were repeated as carried out as carried out for flasks 1-6 in Set 1.

Results

Set 1

Flasks	Initial reading(mL)	Final reading(mL)	Volume of Na ₂ S ₂ O ₃ used (mL)
1	0.0	4.8	4.8
2	4.8	12.7	7.9
3	12.7	23.4	10.7
4	23.4	36.9	13.5
5	0.0	16.3	16.3
6	3.2	30.2	27.0

Set 2 :

Flasks	Initial reading(mL)	Final reading(mL)	Volume of Na ₂ S ₂ O ₃ used (mL)
7	9.2	10.3	1.1
8	12.5	14.1	1.6
9	10.3	12.5	2.2
10	14.1	16.9	2.8
11	5.8	9.2	3.4
12	0.0	5.8	5.8

Question

1. Calculate N for iodine in each flask.

<u>Flask 1</u>

Volume of Na₂S₂O₃ used = 4.8mL Molarity of Na₂S₂O₃ = 0.1M Number of moles of Na₂S₂O₃ = 4.8×0.1/1000 = 4.8 ×10⁻⁴mol 1 mol Na₂S₂O₃ reacts with 0.5 mol I₂. So, 4.8 ×10⁻⁴mol Na₂S₂O₃ reacts with 2.4×10⁻⁴mol I₂. Concentration of I₂(X) =mole/volume = 2.4×10⁻⁴mol/0.05L = 4.8×10⁻³M <u>Flask 7</u> Volume of Na₂S₂O₃ used = 1.1mL Molarity of Na₂S₂O₃ = 0.1M Number of moles of Na₂S₂O₃ = 1.1×0.1/1000 = 1.1×10⁻⁴mol 1 mol Na₂S₂O₃ reacts with 0.5 mol I₂. So, 1.1×10⁻⁴mol Na₂S₂O₃ reacts with 5.5×10⁻⁵mol I₂. Concentration of I₂(C) =mole/volume

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=5.5 \times 10^{-5} \text{ mol}/0.012 \text{ L}
= 4.58 \times 10^{-3}M
N = (X-C)
= (4.8 \times 10^{-3} \text{M} - 4.58 \times 10^{-3} \text{M})
= 1.1 \times 10^{-4}
Flask 2
Volume of Na_2S_2O_3 used = 7.9mL
Molarity of Na_2S_2O_3 = 0.1M
Number of moles of Na_2S_2O_3 = 7.9 \times 0.1/1000
= 7.9 \times 10^{-4} mol
1 mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 0.5 mol I<sub>2</sub>. So, 7.9 ×10<sup>-4</sup>mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 3.95 \times 10^{-4}mol I<sub>2</sub>.
Concentration of I_2(X) =mole/volume
=3.95×10<sup>-4</sup>mol/0.05L
= 7.9 \times 10^{-3} M
Flask 8
Volume of Na_2S_2O_3 used = 1.6mL
Molarity of Na_2S_2O_3 = 0.1M
Number of moles of Na_2S_2O_3 = 1.6 \times 0.1/1000
= 1.6 \times 10^{-4} mol
1 mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 0.5 mol I<sub>2</sub>. So, 1.6 \times10<sup>-4</sup>mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 8\times10<sup>-5</sup>mol I<sub>2</sub>.
Concentration of I<sub>2</sub>(C) =mole/volume
=8×10<sup>-5</sup>mol/0.012L
= 6.67 \times 10^{-3}M
N = (X-C)
= (7.9 \times 10^{-3} \text{M} - 6.67 \times 10^{-3} \text{M})
= 6.15 \times 10^{-4}
Flask 3
Volume of Na_2S_2O_3 used = 10.7mL
Molarity of Na_2S_2O_3 = 0.1M
Number of moles of Na_2S_2O_3 = 1.07 \times 0.1/1000
= 1.07 \times 10^{-3} mol
1 mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 0.5 mol I<sub>2</sub>. So, 1.07 \times 10^{-3}mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 5.35 \times 10^{-4}mol I<sub>2</sub>.
Concentration of I<sub>2</sub>(X) =mole/volume
=8×10<sup>-5</sup>mol/0.012L
= 0.0107M
Flask 9
Volume of Na_2S_2O_3 used = 2.2mL
Molarity of Na_2S_2O_3 = 0.1M
Number of moles of Na_2S_2O_3 = 2.2xo.1/1000
= 2.2 \times 10^{-4} mol
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1 mol Na₂S₂O₃ reacts with 0.5 mol I₂. So, 2.2 ×10⁻⁴mol Na₂S₂O₃ reacts with 1.1×10^{-4} mol I₂. Concentration of I₂(C) =mole/volume =5.35×10⁻⁴mol/0.05L $= 9.17 \times 10^{-3} M$ N = (X-C) $= (0.0107 M - 9.17 \times 10^{-3} M)$ $= 7.65 \times 10^{-4}$ <u>Flask 4</u> Volume of $Na_2S_2O_3$ used = 13.5mL Molarity of $Na_2S_2O_3 = 0.1M$ Number of moles of $Na_2S_2O_3 = 1.35 \times 0.1/1000$ $= 1.35 \times 10^{-3}$ mol 1 mol Na₂S₂O₃ reacts with 0.5 mol I₂. So, 1.35×10^{-3} mol Na₂S₂O₃ reacts with 6.75×10^{-4} mol I₂. Concentration of $I_2(X)$ =mole/volume =6.75×10⁻⁴mol/0.05L = 0.0135M Flask 10 Volume of $Na_2S_2O_3$ used = 2.8mL Molarity of $Na_2S_2O_3 = 0.1M$ Number of moles of $Na_2S_2O_3 = 2.8 \times 0.1/1000$ $= 2.8 \times 10^{-4}$ mol 1 mol Na₂S₂O₃ reacts with 0.5 mol I₂. So, 2.8 ×10⁻⁴mol Na₂S₂O₃ react with 1.4×10⁻⁴mol I₂. Concentration of I₂(C) =mole/volume =1.4×10⁻⁴mol/0.012L = 0.0117 M N = (X-C)= (0.0135M - 0.0117M) $= 9.0 \times 10^{-4}$ Flask 5 Volume of $Na_2S_2O_3$ used = 16.3mL Molarity of $Na_2S_2O_3 = 0.1M$ Number of moles of $Na_2S_2O_3 = 1.63 \times 0.1/1000$ $= 1.63 \times 10^{-3}$ mol 1 mol Na₂S₂O₃ reacts with 0.5 mol I₂. So, 1.63 ×10⁻³mol Na₂S₂O₃ reacts with 8.15×10⁻⁴mol I₂. Concentration of $I_2(X)$ =mole/volume =8.15×10⁻⁴mol/0.012L = 0.0163MFlask 11 Volume of $Na_2S_2O_3$ used = 3.4mL

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Molarity of Na_2S_2O_3 = 0.1M
Number of moles of Na_2S_2O_3 = 3.4 \times 0.1/1000
= 3.4 \times 10^{-4} mol
1 mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 0.5 mol I<sub>2</sub>. So, 3.4 \times 10^{-4}mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 1.7 \times 10^{-4}mol I<sub>2</sub>.
Concentration of I<sub>2</sub>(C) =mole/volume
=1.7×10<sup>-4</sup>mol/0.012L
= 0.0142 M
N = (X-C)
= (0.0163M - 0.0142M)
= 1.05 \times 10^{-3}
Flask 6
Volume of Na_2S_2O_3 used = 27.0mL
Molarity of Na_2S_2O_3 = 0.1M
Number of moles of Na_2S_2O_3 = 2.7 \times 0.1/1000
= 2.7 \times 10^{-3} mol
1 mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 0.5 mol I<sub>2</sub>. So, 2.7 ×10<sup>-3</sup>mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 1.35 \times 10^{-3}mol I<sub>2</sub>.
Concentration of I_2(X) =mole/volume
=1.35×10<sup>-3</sup>mol/0.05L
= 0.027M
Flask 12
Volume of Na_2S_2O_3 used = 5.8mL
Molarity of Na_2S_2O_3 = 0.1M
Number of moles of Na_2S_2O_3 = 5.8 \times 0.1/1000
= 5.8 \times 10^{-4} mol
1 mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 0.5 mol I<sub>2</sub>. So, 5.8 ×10<sup>-4</sup>mol Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 2.9×10<sup>-4</sup>mol I<sub>2</sub>.
Concentration of I<sub>2</sub>(C) =mole/volume
=2.9×10<sup>-4</sup>mol/0.012L
= 0.0242 M
N = (X-C)
= (0.027 M - 0.0242 M)
= 1.4 \times 10^{-3}
```

1. Plot amount of iodine adsorbed (N) versus balance concentration of solution at equilibrium to obtain adsorption isotherm.

Balance concentration of solution (C)	Amount of iodine adsorbed (N)
4.58×10 ⁻³ M	1.1 ×10 ⁻⁴
6.67×10 ⁻³ M	6.15 ×10 ⁻⁴
9.17×10 ⁻³ M	7.65 ×10-4
0.0117 M	9.0 ×10 ⁻⁴

0.0142 M	1.05 ×10 ⁻³
0.0242 M	1.4 ×10 ⁻³

Conclusion

The rate of adsorption increases with increasing concentration of absorbate.

Aim: To determine the rate constant for the acid-catalyzed hydrolysis of methyl acetate.

Requirement:

*Apparatus:*thermostat, reagent bottles, conical flask, burette, pipette *Chemicals:* Methyl acetate, HCl, NaOH, 0.1N standard Oxalic acid, phenolphthalein, thermostat, reagent bottles, conical flask, burette, pipette, ice.

Theory:

Methyl acetate undergoes hydrolysis, in the presence of an acid (HCl), to give acetic acid and methyl alcohol.

$^{H^+}$ CH₃COOCH₃ + H₂O \rightarrow CH₃COOH + CH₃OH

In the presence of an acid, this reaction should be of second order, since two molecules are reacting. But, it is found to be first order. This may be explained in the following way:

The rate of the reaction is given by

 $\frac{dx}{dt} = k'[CH_3COOCH_3][H_2O]$

where k' is the rate constant(or specific rate constant).

Since water is present in large excess, its active mass (molar concentration) virtually remains constant during the course of the reaction. Therefore, its active mass gets included in the constant, and the above equation reduces to:

$$\frac{dx}{dt} = k_1 [CH_3COOCH_3]$$

Thus, the rate of the reaction is determined by one concentration term only (that is, by a single power of the concentration term only).Hence, the reaction is first order. Such reactions are also referred to as pseudo first order reactions.The progress of the reaction (hydrolysis of ester) is followed by removing a definite volume of the reaction mixture, at definite intervals of time, cooling it in ice, and titrating the acetic acid formed against alkali, which has already been standardized.The amount of alkali used is equivalent to the total amount of hydrochloric acid present originally and the amount of acetic acid formed in the reaction. The amount of acetic acid formed (x), at definite intervals of time (t), can be obtained. The amount of acetic acid formed, at the end of the reaction, is equivalent to the initial concentration (a) of the ester. Suppose the volumes of the sodium hydroxide solution (titre value) required for neutralization of 5 mL of the reaction mixture are:

- (i) at the commencement of the reaction is V_o
- (ii) after time (t) is V_t
- (iii) at the end of the reaction is V_{∞}

x (amount of acetic acid formed after time) is proportional to $(V_t - V_o)$. a (initial concentration of ester) is proportional to $(V_{\infty} - V_o)$. [a - x] (conc. of ester present after time t) is proportional to $(V_{\infty} - V_o) - (V_t - V_o) = (V_{\infty} - V_t)$

The first order rate expression given by: $k_1 = \frac{2.303}{t} \log \frac{V \otimes -V \circ}{V \otimes -V \circ}$

Hence, the rate constant (k_1) could be calculated.

Procedure

Step I: Standardization of NaOH using standard Oxalic acid(0.1N)

1.10mL of given 0.1N standard Oxalic acid is pipetted out into a 100mL conical flask.

- 2. This solution is titrated against the given unknown concentration of NaOH using phenolphthalein indicator until the end point is colorless to pale pink.
- 3. Tabulate the values and repeat the titration for concurrent readings and determine the unknown concentration of supplied NaOH solution.

Table 1

S. No.	Volume of Oxalic acid taken	Burette reading		Volume of NaOH
	(mL)	Initial	Final	used (mL)
1				
2				

 $N_1 V_1 = N_2 V_2$ (oxalic acid) (NaOH)

Concentration of NaOH, N₂ = _____

Step II: Standardization of HCl using NaOH solution.

1.20 mL of given HCl is pipetted out into a 100mL conical flask.

- 2. This solution is titrated against the NaOH using phenolphthalein indicator until the end point is colorless to pale pink.
- 3. Tabulate the values and repeat the titration for concurrent readings and determine the unknown concentration of supplied HCl solution.

Table Z				
S. No.	Volume of HCl taken (mL)	Burette reading		Volume of NaOH
		Initial	Final	used (mL)
1				
2				

 $\begin{array}{ll} N_2V_2 &=& N_3V_3 \\ (NaOH) && (HCl) \end{array}$

Concentration of HCl, N₃ = _____

T-1-1- 0

Step III: Determination of rate constant (k₁) for the acid-catalyzed hydrolysis of methyl acetate.

- 1. 100 mL of given HCl (whose strength is determined in step II) solution is taken in a stoppered reagent bottle.
- 2. 5 mL of methyl acetate solution is added to the HCl solution. Note the time when half of the methyl acetate solution is added. The mixture is shaken well.
- 3. Pipette out 5 mL of the reaction mixture and discharge it into 50 mL of ice cold water kept in a conical flask.
- 4. Titrate the reaction mixture against NaOH solution using phenolphthalein as indicator. This titre value corresponds to V_0 .
- 5. Steps 3 and 4 are repeated at intervals of 5, 10, 15, 20, 30, 45, 60 minutes. Each titre value corresponds to $V_t\,.$
- 6. The remaining solution is taken in a stoppered conical flask and heated to 60°C, and kept at this temperature for 5 minutes.
- 7. The solution is allowed to cool to room temperature.
- 8. Repeat Steps 3 and 4. This titre value corresponds to V_{∞} till concurrent values are obtained.
- 9. Plot a graph of log (V_{∞} - V_t) versus time (t) and determine the slope. Report the theoretical and graphical value of rate constant (k_1).

Table 3

	Time	Volume	Bu Rea	arette adings	Volume		log(V∞-Vt)	$k_1 = \frac{2.303}{\log 2000} \log \frac{V \infty - V_0}{\log 2000}$
S.No	(min)	solution taken (mL)	Initial	Final	NaOH consumed (mL)	(V∞-V _t) (mL)		$(\min^{-1})^{1} \nabla \infty - Vt$
1	0 (V ₀)							
2	5 (V _{t1})							
3	10 (V _{t2})							
4	15 (V _{t3})							
5	20 (V _{t4})							
6	30 (V _{t5})							
7	45 (V _{t6})							
8	60 (V _{t7})							
9	V∞							

Observations: Room Temperature = °C;

 V_{∞} = mL ; V_{o} = mL ; $(V_{\infty}-V_{o})$ = mL ; $\log(V_{\infty}-V_{o})$ = Mean Value of Rate Constant (k₁) = _____

Results

Strength of NaOH Solution =..... Strength of HCl Solution = Rate Constant (k1) for the acid-catalyzed hydrolysis of methyl acetate (CH₃COOCH₃) =

Aim: To detect the extra elements (Nitrogen, Sulphur and Halogens) present in the given organic compound.

Requirements:

- a) Apparatus: Ignition tubes or Fusion tubes, China dish, burner, filter paper and test tubes.
- b)Chemicals : Ferrous Sulphate solution, Ferric Chloride, HCl, Sodium Nitroprusside, AgNO₃, conc. HNO₃, Lead acetate, NH₄OH and Sodium metal.

Preparation of Lassaigne's Extract or sodium extract:

Take a freshly pea size cut piece of sodium metal and dry it by pressing between the folds of a filter paper. Place the metal in the dry ignition tube. Heat the tube slowly when it melts to a shining globule. Remove it from the flame and add small amount of the compound. Heat the tube on a flame until it becomes red hot. Keep it in the red hot condition for 1-2 minutes and then plunge into 20 mL of distilled water kept in a china dish. Boil the contents of the china dish for 10 minutes and filter. The filtrate is known as Sodium Extract (S.E.) or Lassaigne's Extract (L.E.). The solution should be transparent. Use the filtrate for testing the extra elements.

Chemical Equation:

 $\begin{array}{ll} Na + C + N & \longrightarrow NaCN \\ 2Na + S & \longrightarrow Na_2S \\ Na + X & \longrightarrow NaX (X = Cl, Br, I) \\ Na + C + N + S & \longrightarrow NaSCN \end{array}$

Test for Nitrogen:

Experiment	Observation	Inference
FeSO₄ test: To the sodium extract, add solid Ferrous Sulphate. Boil the mixture gently for one minute and acidify with dil. H ₂ SO ₄ .	A blue precipitate (Prussian blue) or blue colors indicate the presence of nitrogen.	Nitrogen present

Chemical reaction:

 $6 \text{ NaCN} + \text{FeSO}_4 \rightarrow \text{Na}_4[\text{Fe}(\text{CN})_6] + \text{Na}_2\text{SO}_4$ Sodium Ferrocyanide $3 \text{ Na}_4[\text{Fe}(\text{CN})_6] + 2 \text{ Fe}_2(\text{SO}_4)_3 \rightarrow Fe_4[\text{Fe}(\text{CN})_6]_3 + 6 \text{ Na}_2\text{SO}_4$ Ferric Ferrocyanide (Prussian blue) Test for Nitrogen and Sulphur present together:

Experiment	Observation	Inference
FeCl ₃ test : Acidify the sodium extract (2 mL) with dil. HCl (5-6 drops) and treat with ferric chloride solution (2-3 drops).	Appearance of a blood red color	Nitrogen and Sulphur present

Chemical Equation:

3 NaSCN + FeCl₃ \rightarrow [Fe(SCN)(H₂O)₅] + 3 NaCl Ferric thiocyanate (blood red)

Test for Sulphur:

Experiment	Observation	Inference
i) Sodium Nitroprusside test: To the L.E. add a few drops of freshly prepared aqueous solution of sodium nitroprusside.	A deep reddish violet coloration or purple color	Sulphur present
ii) Lead acetate test: Acidify the sodium extract (2-3 mL) with dil. acetic acid (2-3 mL) and add a few drops of lead acetate solution.	Appearance of black ppt.	Sulphur present

Chemical reaction:

i)	$Na_2S + Na_2[Fe(CN)_5NO] \rightarrow Na_4[Fe(CN)_5NOS]$
	Sod. nitroprusside Sod. sulphonitroprusside (reddish violet)
ii)	$Na_2S + (CH_3COO)_2Pb \rightarrow PbS\downarrow + 2CH_3COONa$
	black ppt

Test for Halogens:

Experiment	Observation	Inference
Silver Nitrate Test: Acidify the sodium extract (2-3 mL) with dil. Hydrochloric acid (5-6 drops) and	a) White ppt. is formed which is soluble in NH4OH.	Cl- present
treat with sodium chloride solution (2-3 drops).	 b) A pale yellow ppt. is formed which is soluble in excess of NH₄OH. 	Br- present
	c) A yellow ppt. which is insoluble in NH₄OH.	I- present
Layer Test: To the sodium extract	If organic layer attains	
(2 mL) add conc. Nitric acid (1 mL). Heat the mixture for a few seconds.	a) Violet color	I- present
Add CCl ₄ or CHCl ₃ (1 mL) and shake well.	b) Orange brown	Br− present
		Cl- present
	c) Silver nitrate test: a white ppt.	

Chemical reacton:

 $NaX + AgNO_3 \rightarrow AgX \downarrow + NaNO_3 \qquad (X = Cl, Br, I)$ ppt.

Precaution:

- 1. **Never throw sodium metal in the sink**. Do not touch the sodium with fingers; handle it with forceps.
- 2. Laboratory Coat must be worn at all time during the lab period.
- 3. When you are heating a test tube, never points its mouth towards yourself or your neighbours.

Result: In the given organic compound extra element ------is/are present.

Aim: To detect the functional group Carboxylic acid (-COOH) present in the given organic compound.

Requirements:

- a) Apparatus: China dish, burner, filter paper and test tubes.
- b) **Chemicals:** given organic compound, blue litmus solution, sodium bicarbonate, ethanol and conc. H_2SO_4 .

Procedure:

Ex	periment	Observation	Inference
i)	Litmus test: Add blue litmus (1-2 drops) to an aqueous solution of acid (2 mL).	Appearance of a red color	Carboxylic acid may be present
ii) iii	 Sodium bicarbonate test: To a saturated solution of sodium bicarbonate in water (1 mL) add a small quantity of the substance to it. Ester test: Heat a mixture of small quantity of compound, ethanol (1-2 mL) and conc. H₂SO₄ (2-3 drops) in a dry test tube in a water bath. 	Strong effervescences with the evolution of CO ₂ gas which turns lime water milky	Carboxylic acid present
		A fruity smell of ester	Carboxylic acid present

Chemical Equation:

- ii) NaHCO₃ + RCOOH \rightarrow RCOONa + H₂O + CO₂ Sod. bicarbonate
- iii) RCOOH + R'OH + conc. $H_2SO_4 \rightarrow RCOOR' + H_2O$

Result: In the given organic compound extra element ------is/are present.

Aim: To detect the functional group Phenol present in the given organic compound.

Requirements:

- a) Apparatus: China dish, burner, filter paper and test tubes.
- b) Chemicals: given organic compound, blue litmus solution, ferric chloride, cerric ammonium nitrate solution, sodium nitrite solution and conc. sulphuric acid.

Procedure:

Ex	periment	Observation	Inference
i)	Litmus test: Shake a pinch of given organic compound with 2-3 mL distilled water. Add 2-3 drops of blue litmus solution.	Appearance of a red color	Phenol may be present
ii)	Ferric Chloride test: Take about 5 mL FeCl ₃ solution in a dry test tube. Add into it 2-3 drops of compound.	Appearance of dark blue, green, purple or red colored solution	Phenol present
iii)	Cerric ammonium nitrate test: Take about 1 mL of Cerric ammonium nitrate solution in a dry test tube. Add to it a pinch of the compound.	Appearance of green or brown ppt.	Phenol present
iv)	Libermann's test: Take about 0.1 gm of the compound in a dry test tube and add 1 mL conc. H_2SO_4 to it. Shake and warm the test tube. Cool and then add a pinch of NaNO ₂ .	A deep blue or green color which turns red on dilution	Phenol present
		with water carefully	

Chemical Equation:

ii) $6 C_6H_5OH + FeCl_3 \rightarrow H_3[Fe(OC_6H_5)_6] + 3 HCl$

Purple iii) $(NH_4)_2 [Ce(NO_3)_6] + C_6H_5OH \rightarrow (NH_4)_2 [Ce(OC_6H_5)(NO_3)_5] + HNO_3$ Green or brown ppt

Result: In the given organic compound functional group ------is present

Experiment: 7

Aim: To detect the functional group Alcohol (-OH) present in the given organic compound.

Requirements:

- a) Apparatus: China dish, burner, filter paper and test tubes.
- b) Chemicals: given organic compound, cerric ammonium nitrate solution and acetic acid.

Procedure:

Experiment	Observation	Inference
I) Cerric ammonium nitrate test: Take	Appearance of green or	Alcohol present
about 1 mL of Cerric ammonium nitrate solution in a dry test tube. Add to it a pinch of the compound.	brown ppt.	
 II) Ester test: Heat a mixture of small quantity of compound, acetic acid (1 mL) and conc. H₂SO₄ (2-3 drops) in a dry test tube in a water bath. 	A fruity smell of ester will be observed.	Alcohol present

Chemical Equation:

I)
$$(NH_4)_2[Ce(NO_3)_6] + ROH \rightarrow (NH_4)_2[Ce(OR)(NO_3)_5] + HNO_3$$

Green or brown ppt.

II)
$$CH_3COOH + ROH + conc. H_2SO_4 \rightarrow CH_3COOR + H_2O$$

Precaution:

1. Laboratory Coat must be worn at all time during the lab period.

- 2. When you are heating a test tube, never points its mouth towards yourself or your neighbours.
- 3. Do not smell test tube directly.

Result: In the given organic compound extra element -----is/are present.

Aim: To detect the functional group Aldehyde and Ketone (Carbonyl compounds, >C=O) present in the given organic compound.

Requirements:

- a) Apparatus: China dish, burner, filter paper and test tubes.
- b) Chemicals: given organic compound, 2,4-dinitrophenylhydrazine, Schiff's reagent, Tollen's reagent, Fehling solution A, Fehling solution B, m-dinitrobenzene, sodium nitroprusside solution, NaOH.

Procedure:

Common test for carbonyl compounds:

Experiment	Observation	Inference
2,4-dinitrophenylhydrazine test: Take	Appearance of orange or	Carbonyl group
about 3 mL of 2,4-dinitrophenylhydrazine	yellow ppt.	(>C=O) present
reagent in a test tube. Add it to small quantity		
of given organic compound and shake well.		

Chemical Reaction:



Orange or yellow ppt.

Tests for aldehydes (-CHO):

Experiment	Observation	Inference
 i) Schiff's reagent test: Take 2 mL of aq. Solution of organic comp. in a test tube and add about 3 mL of Schiff's reagent to it. Shake well and wait for minute. 	Appearance of violet or purple colour	Aldehyde present
 ii) Fehling's test: Take 2 mL of aq. Solution of compound and add 1 mL Fehling's solution. Warm the content in a water bath for a few minutes. 	Appearance of red ppt.	Aldehyde present

iii) Tollen's test: Take about 2 mL of Tollen's	Appearance of silver	Aldehyde present
reagent in a test tube. Add small quantity	mirror or formation	
of organic compound to it.	of grey black ppt.	

Chemical Reaction:

i) Schiff's test:



Colourless

ii) Fehling test: HCHO + 2 CuO \rightarrow HCOOH + Cu₂O Cupric oxide cuprous oxide (red ppt.)

iii) Tollen's test: $\text{RCHO} + \text{Ag}_2\text{O} \rightarrow \text{RCOOH} + 2\text{Ag}$ on test tube surface

Test for ketones (R₂C=O)

Ex	periment	Observation	Inference
i)	m-dinitrobenzene test: Take 4-5 drops	Appearance of violet color	Ketone group
	of the given organic compound in a dry	which fades on standing	present
	test tube. Add about 0.1 gm of powdered		
	m-dinitro benzene followed by 5 mL of		
	NaOH solution and shake well.		
		Appearance of wine red	Ketone group
ii)	Sodium nitroprusside test: Take 2 mL	colour	present
	of freshly prepared sodium nitroprusside		
	solution in dry test tube. Add small		
	quantity of given organic compound.		
	Shake and add NaOH solution dropwise.		

*benzophenone does not give m-dinitrobenzene test

Chemical Equation:

m-dinitrobenzene test:



ii) Sodium nitroprusside test : $CH_3COCH_3 + OH^- \rightarrow CH_3COCH_2^- + H_2O$

 $[Fe(CN)_5NO]^{2-} + CH_3COCH_2^- \rightarrow [Fe(CN)_5NO.CH_3COCH_2]^{3-}$

wine red

Result: In the given organic compound functional group -----is/are present.

Aim: To detect the functional group Ester (-COOR) present in the given organic compound.

Requirements:

- a) Apparatus: China dish, burner, filter paper and test tubes.
- b) Chemicals: given organic compound, Phenolphthalein, Sodium hydroxide, Hydroxyl amine hydrochloride, HCl, Ferric chloride solution

Procedure:

Experiment	Observation	Inference
i) Phenolphthalein test: To organic compound add 2-3 drops of a dilute solution of NaOH and one drop of phenolphthalein and heat it.	Pink colour is discharged	Ester group is present
ii) Hydroxamic acid test (Feigi test): Dissolve a small amount of organic compound in 5 mL of NaOH solution. Add to it 0.2 g of hydroxyl amine hydrochloride. Boil for two minutes, cool, acidify with HCl and add 2-3 drops of ferric chloride solution.	Appearance of violet, green, blue or red color	Ester group is present

Chemical reaction:

i) $CH_3COOC_2H_5 + H_2O \rightarrow CH_3COOH + C_2H_5OH$

 $CH_{3}COOH + NaOH \rightarrow CH_{3}COONa + H_{2}O$

ii) RCOOR' + NH₂OH \rightarrow R'OH + RCONHOH



RCONHOH + $FeCl_3 \rightarrow Magenta coloured complex$

Result: In the given organic compound functional group _______is present.

Aim: To detect the functional group Acid amide (-CONH₂) present in the given organic compound.

Requirements:

- a) Apparatus: China dish, burner, filter paper and test tubes.
- b) Chemicals: given organic compound, Sodium hydroxide, Nessler's reagent, dil. HCl, 10% NaNO₂

Procedure

Experiment		Observation	Inference
i)	Sodium hydroxide test: Heat a pinch of organic compound with 15% NaOH in water.	Ammonical smell turning moist turmeric paper brown and giving brown ppt. with Nessler's reagent	Amide group present
ii)	Nitrous acid test: Dissolve a pinch of organic compound in about 5 mL of dil. HCl and add a few drops of 10 % NaNO ₂ into it.	Brisk effervescence due to evolved nitrogen	Amide group present
<u>Co</u> 1	nfirmation test for Urea	Formation of a purple colour	Urea (amide group)
iii)	Biuret test: Heat the given compound (0.2 g) in a dry test tube till the melted compound solidifies and evolution of NH ₃ ceases, cool, dissolve the solid in dil. NaOH and add a dilute solution of copper sulphate (1-2 drops).		confirmed

Chemical Reaction:

i) RCONH₂ + NaOH \rightarrow RCOONa + NH₃ + H₂O

ii) NaNO₂ + HCl \rightarrow NaCl + HNO₂

 $RCONH_2 + HNO_2 \rightarrow RCOOH + N_2\uparrow + H_2O$

Biuret test

 $2 \text{ NH}_2\text{CONH}_2 \xrightarrow{dry\Delta} \text{ NH}_3 + \text{NH}_2\text{CONHCONH}_2 \xrightarrow{coppersuiphate} \rightarrow \text{Purple coloured complex}$ Biuret

Result: In the given organic compound functional group _______is present.

Aim: To detect the functional group Amines (-NH₂) present in the given organic compound.

Requirements:

- a) Apparatus: China dish, burner, filter paper and test tubes.
- b) Chemicals: given organic compound, red litmus, conc. HCl, CHCl₃, 10% alc. solution of KOH, sodium nitrite, β naphthol, Tollen's reagent, 10% CaCl₂ solution and zinc metal.

Procedure: Test for amine (-NH₂) group

Expe	eriment	Observation	Inference
i)	Litmus test: Take small quantity of organic	Red litmus paper	amine may be
	compound into a dry test tube. Dip red litmus paper in to test tube.	turns blue	present
	Contribution on icogramida toot. Taka amall	A very unpleasant	amine group
11)	quantity of substance in test tube and add 1	smell of isocyanides	present
	mL conc. HCl and few drops of CHCl ₃ to it.		
	Then add about 2 mL of 10 % alcoholic		
	solution of KOH and warm.	Formation of	amine group
iii)	Dye test: Take small amount of organic	orange- red dye	is confirmed
	compound and add 1 mL water followed by 1		
	mL of NaNO ₂ solution and then cool it in ice		
	bath. In another test tube cool about 2 mL		
	alkaline solution of β -naphthol and mix the		
	two solutions		

Chemical reaction:

 $RNH_2 + CHCl_3 + 3 \text{ KOH (alcohlic)} \rightarrow Amine$

RNC + 3 KCl + 3 H₂O isocyanide

Dye test:

 $C_6H_5NH_2 \ \ \text{+}\ HCl {\rightarrow} \ \ C_6H_5N^+H_3Cl^-$

 $NaNO_2 + HCl \rightarrow HNO_2 + NaCl$

 $C_6H_5N^+H_3Cl^-$ + ice $O_2 \rightarrow C_6H_5N_2^+Cl^-$ Dizonium chloride



orange-red **Result**: In the given organic compound functional group -----is present.

Aim: To demonstrate the effect of sodium thiosulfate concentration on the rate of reaction of sodium thiosulfate with hydrochloric acid.

Apparatus: 05Beakers 50-mL, graduated cylinders, 5 stirring rods, stopwatch, clamp and permanent marker

Chemicals: HCl (2 M), sodium thiosulfate solution (0.15 M), distilled or deionized water

Theory: The reaction of sodium thiosulfate with hydrochloric acid. The reaction, which produces solid sulfur, will be followed by measuring the time needed for the reaction mixture to become opaque. The results will be analyzed graphically to determine the relationship between the reactant concentration and the rate.

Sodium thiosulfate reacts with hydrochloric acid to form sulfur and sulfur dioxide

 $Na_2S_2O_3(aq) + 2HCl(aq) \rightarrow S(s) + SO_2(g) + 2NaCl(aq) \qquad \dots eq 1$

The effect of concentration can be analyzed by graphing the concentration of $Na_2S_2O_3$ as a function reaction time. A plot of concentration versus time gives a curved line, which levels off as it approaches the x-axis—the reaction slows down as the reactant concentration decreases. The rate of a reaction is inversely proportional to reaction time.

Procedure: 1. Label five 100-mL beakers 1–5 and clean the bottom of each beaker.

2. Draw a large "X" across the bottom on the outside of each beaker.

3. Using separate graduated cylinders for the solution and water, measure and add the required amounts of 0.15 M sodium thiosulfate and distilled water to each beaker. Be as precise as possible.

4. Ask students calculate the final concentration of sodium thiosulfate in each beaker 1–5.

Beaker	1	2	3	4	5
Volume of	50 mL	40 mL	30 mL	20 mL	10 mL
$Na_2S_2O_3(0.15 M)$					
Volume of water	0 mL	10 mL	20 mL	30 mL	40 mL

5. Record the following information in a data table: Beaker, volume of Na₂S₂O₃ solution, volume

of distilled water, concentration of Na₂S₂O₃, reaction time (sec),

6. Measure 5.0 mL of 2 M hydrochloric acid into each of five 10-mL graduated cylinders.

7. Starting with beaker #1, carefully add the HCl all in one pour to the sodium thiosulfate solution. Stir the solution once with a stirring rod and immediately start timing.

8. Stop timing when the black "X" is no longer visible. Record the reaction time in seconds in the data table.

9. Repeat steps 7 and 8 with beakers 2–5.

10. Plot concentration vs. time on separate graphs.

Observation Table

Beaker	Volume of Na ₂ S ₂ O ₃ (mL)	Volume of water	Conc of Na ₂ S ₂ O ₃	Reaction time in sec
1	50	0		
2	40	10		
3	30	20		
4	20	30		
5	10	40		

The student can be able to plot a graph as seen below.



Aim: To Prepare Colloidal Solution (sol) of starch

Apparatus

Beakers (250 mL and 50 mL), glass rod, funnel, filter-paper, pestle and mortar, tripod stand, wire-gauze and burner.

Chemicals: Starch (500 mg) and distilled water.

Theory

Starch forms a lyophilic sol when water is used as the dispersion medium. The formation of sol is accelerated by heating. The starch sol can be prepared by heating starch and water at about 100°C. It is quite stable and is not affected by the presence of any electrolytic impurity.

Procedure: 1. Take 500 mg of starch in a mortar and add few mL of distilled water. Grind the starch to make a thin paste and transfer this paste to a 50 mL beaker.

2. Take about 100 mL of distilled water in a 250 mL beaker and heat the beaker so that water starts boiling.

3. Pour the paste slowly with stirring into boiling water in the beaker.

4. Continue boiling for about 10 minutes and there allow the beaker to cool.

5. Filter the contents of the beaker through a filter-paper, fixed in a funnel. Label the filtrate 'Starch Sol'