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Maharashtra Institute of Technology, Aurangabad
Department of Plastics and Polymer Engineering
LABORATORY MANUAL

ORGANIC CHEMISTRY MANUAL

SEMESTER-I

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Maharashtra Institute of Technology, Aurangabad

Department of Plastics and Polymer Engineering

LABORATORY MANUAL

ACADEMIC YEAR: 2019-20 **PART:I**

COURSE:Organic Chemistry **COURSE COORDINATOR:Ms.A.S.Dutta**

Experiment No.-1

1.Aim: To find out the percentage purity of given Phenol sample.

2.Theory:Phenol on bromination gives tribromophenol bromide. When potassium iodide is added to it, it gets converted to tribromophenol. This which can be estimated by treating it with sodium thiosulphate. Hence by knowing the amount of bromine used the amount of phenol can be estimated.

3.Apparatus:

a.Conical flask
b.Beaker
c.Burette
d.Pipette
e.Measuring cylinder
f.Test tube

4.Chemicals:

- **a.** **(**Potasium bromide and Potassium bromate)0.1N,
- **b.** Conc. Hydrochloric acid,
- **c.** Sodium thiosulphate,0.1N,
- **d.** Potassium iodide 10%,
- **e.** 2% Phenol solution
- **f.** Starch

5.Procedure:

1.Pipette out 10 ml phenol in the conical flask and add it 5 ml of Conc.

2.Hydrochloric acid, and 25 ml of **(**Potasium bromide and Potassium bromate) 0.1N solution and 25 ml of water.
3.Allow it to stand for 10 minutes.
4.Then add25 ml of Potassium iodide 10% solution.
5. Titrate the librated iodine against Sodium thiosulphate 0.1N solution using starch as an indicator.
6.End point is blue to colorless.
7.Note down the burette reading as sample burette reading .
8.Carry out blank titration and note down the burette reading.

6.Observations:

Sr.No.	Sample burette reading (ml)	Blank burette reading(ml)
1.		
2.		
3.		
4.	Mean burette reading ---------- ml	Mean burette reading ---------- ml

Sample Burette reading (V1): ml

Blank burette reading(V2) : ml

7.Sample Calculation:

6g equivalent of sodium thiosulphate == 1g mole of phenol

6000 ml of 1N sodium thiosulphate =94 g of phenol

(V2-V1) ml of 0.1 sodium thiosulphate = 94x (V2-V1)/6000

% purity = (V2-V1) N x mol wt. x 100/ 2000 x No. of Bromine atoms substituted

8.Result: The percentage purity of phenol is ----------

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9.Conclusion: Phenol analysed has ---------- percentage purity.

10.Applications:

11.Assignment Questions:

a. Explain in detail electrophilic substitution reactions of benzene ring of phenol.
b. Explain the reactions involving OH group of phenol.
c. Explain various methods of preparation of phenol.

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ACADEMIC YEAR: 2019-20 **PART:I**

COURSE:Organic Chemistry **COURSE COORDINATOR:Ms.A.S.Dutta**

Experiment No.-2

1.Aim: To find out the percentage purity of given Styrene sample.

2.Theory: The estimation is based on the fact that on treating with brominating mixture it gives tri bromo derivative. The reaction mixture is treated with Potassium iodide when unreacted bromine reacts with iodine to give iodine which can be estimated by treating it with sodium thiosulphate. Hence by knowing the amount of bromine used the amount of Styrene can be estimated;

4g equivalent of sodium thiosulphate == 1g mole of styrene

4000 ml of 1N $Na_2S_2O_3$ sodium thiosulphate =104 g of styrene

3.Apparatus:

a.Conical flask
b.Beaker
c.Burette
d.Pipette
e.Measuring cylinder
f.Test tube

4.Chemicals:

- **a.** **(**Potasium bromide and Potassium bromate)0.1N,
- **b.** Conc. Hydrochloric acid,
- **c.** Sodium thiosulphate,0.1N,
- **d.** Potassium iodide 2 0%,
- **e.** 2% styrene solution,
- **f.** Starch

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5.Procedure:

1. Pipette out 10 ml styrene in the conical flask and add it 5 ml of Conc. Hydrochloric acid, and 25 ml of **(**Potasium bromide and Potassium bromate) 0.1N solution from burette till yellow color persists.
2. Note down the volume.
3.Shake the contents add 5 ml of 20% Potassium iodide solution.
4.Allow it to stand for 10 minutes.
5.Titrate it against Sodium thiosulphate solution using starch as an indicator at the end.
6.Note down the burette reading as sample burette reading.
7.Carry out blank titration and note down the burette reading.

6.Observation:

Sr.No.	Sample burette reading (ml)	Blank burette reading(ml)
1.		
2.		
3.		
4.	Mean burette reading ---------- ml	Mean burette reading ---------- ml

Sample Burette reading (V1): ml

Blank burette reading(V2) : ml

7.Sample Calculation:

4000 ml of 1N $Na_2S_2O_3$=104 g of styrene

Blank reading –Sample reading ……?

104 x $(V_2-V_1)/4000$

% OF PURITY = (V_2-V_1) NxM X 2000x Z

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8.Result: The percentage purity of styrene is ----------

9.Conclusion: Styrene analysed has ---------- percentage purity.

10.Applications:

11.Assignment Questions:

a.Explain in brief chemical properties of styrene.
b.Explain briefly the manufacturing process of styrene with a neatly labeled diagram.

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ACADEMIC YEAR: 2019-20 **PART:I**

COURSE:Organic Chemistry **COURSE COORDINATOR:Ms.A.S.Dutta**

Experiment No.-3

1.Aim: To determine the iodine value of the given sample.

2.Theory: The iodine number is numerically equal to the grams of iodine absorbed by 100 gram of the sample or the percentage by weight of iodine absorbed by the sample.The amount of unsaturation is designated as the iodine number.

3.Apparatus:

a.Conical flask
b.Beaker
c.Burette
d.Pipette
e.Measuring cylinder
f.Test tube

4.Chemicals:

- **a.** Hanus solution(Dissolve 13.2 gram of iodine and 3 ml of bromine in one litre of glacial acetic acid)
- **b.** Potassium iodide 10%,
- **c.** Carbon tetrachloride or Chloroform,
- **d.** Starch
- **e.** Linseed or Castor oil
- **f.** Sodium thiosulphate 0.1N

Following data shows the approximate value of iodine number and the amount of sample and Hanus solution to be taken:

Iodine number	10	50	100	150	200	300
Sample in gram	20	0.4	0.2	0.2	0.2	0.1
Hanus solution in ml	25	25	25	40	50	40

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5.Procedure:

1. Weigh the sample and add to it 10 ml of Carbon tetrachloride or Chloroform.
2. Add required amount of Hanus solution from burette.
3. Stopper the flask ,shake well and allow it to stand for 30 minutes.
4. Add 25 ml of 10% Potassium iodide to it.
5. Titrate it against Sodium thiosulphate solution using starch as an indicator at the end.
6. Note down the burette reading as sample burette reading.
7. Carry out blank titration and note down the burette reading.

6.Observation:

Sr.No.	Sample burette reading (ml)	Blank burette reading(ml)
1.		
2.		
3.		
4.	Mean burette reading ---------- ml	Mean burette reading ---------- ml

Sample Burette reading (V1): ml

Blank burette reading(V2) : ml

7.Sample Calculation:

$$\text{Iodine number} = \frac{\text{Blank reading} - \text{Sample reading} \times N \times 12.7}{\text{Weight of the sample}}$$

8.Result: The iodine value of given sample is ----------

9.Conclusion: The oil sample analysed has ---------- iodine value.

10.Applications:

11.Assignment Questions:

a. Explain in brief the importance of iodine value analysis.
b.Enlist the list of compounds that can be analysed using iodine value and also justify the reason for the same for each compound.

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ACADEMIC YEAR: 2019-20 **PART:I**

COURSE:Organic Chemistry **COURSE COORDINATOR:Ms.A.S.Dutta**

Experiment No.-4

1.Aim: To determine molecular weight of given organic compound by acid base titration method.

2.Theory: The molecular weight of many carboxylic acids which are freely soluble in cold water *(i.e.*, chiefly the aliphatic acids) can readily be obtained by titrating a known weight of the acids in aqueous solution with standard sodium or potassium hydroxide solution, using phenolphthalein as an indicator. To avoid the use of unduly large quantities of the acid, it is advisable to use caustic alkali solution, and in order to obtain a sharp end-point, this alkali solution should be prepared from CO2-free water and pure (ethanol-washed) caustic alkali free from carbonate.

3.Apparatus:

a.Conical flask
b.Beaker
c.Burette
d.Pipette
e.Measuring cylinder
f.Test tube

4.Chemicals:

- **a.** Sodium hydroxide or Potassium hydroxide
- **b.** Phenolphthalein indicator
- **c.** Succinic acid
- **d.** Adipic acid

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5.Procedure:

1. Weigh out accurately about 2-5 gram of pure powdered succinic acid, transfer to a 100 ml. graduated flask, dissolve in distilled water, make the solution up to the graduation mark and mix well.
2. Now, by means of a pipette, transfer 25 ml. of the solution to a 150 ml. conical flask, add a drop of phenolphthalein solution and titrate with M/2 sodium hydroxide or potassium hydroxide solution to obtain consistent results.
3. If the acid had been monobasic, then clearly the molecular weight would have been the weight in grams which neutralises 1000 ml. of normal alkali solution.
4.The molecular weight of the dibasic succinic acid is the weight which neutralises 2000 ml. of the normal alkali solution.
5. Note down the burette readings.

6.Observations:

Sr.No.	Sample burette reading (ml)
1.	
2.	
3.	
4.	Mean burette reading ---------- ml

Sample Burette reading (V1): ml

7.Sample Calculation:

1.Weight of succinic acid taken = z gram
2.Solution made up to 100 ml.
3.25 ml. of this solution required = Mean burette reading(MBR) in ml. M/2 Sodium hydroxide solution
4.100ml of this solution requires = MBR x 4 ml.
5. M/2 Sodium hydroxide solution = MBR x 2 ml. M. Sodium hydroxide solution
6 z g- succinic acid (dibasic) are equivalent to MBR x 2 ml. M. Sodium hydroxide solution
7. So z x 2000/ MBR x 2 ml of succinic acid (dibasic) are equivalent to 2000 ml. M.NaOH
= ----------

Theoretically molecular weight of succinic acid, ($C_4H_6O_4$) =118.

8.Result: The molecular weight of given dibasic acid sample is ----------

9.Conclusion: The practical molecular weight is ---------- .

10.Applications:

11.Assignment Questions:

a. Explain in detail the procedure of calculating molecular weight of monobasic acid.
b. Explain in brief the methods of preparation, physical and chemical properties of
   i.Succinic acid
   ii.Adipic acid

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ACADEMIC YEAR: 2019-20 **PART:I**

COURSE:Organic Chemistry **COURSE COORDINATOR:Ms.A.S.Dutta**

Experiment No.-5

1.Aim: To determine formaldehyde content using sodium sulphite.

2.Apparatus:

a.Conical flask
b.Beaker
c.Burette
d.Pipette
e.Measuring cylinder
f.Test tube

3.Chemicals:

- **a.** 2N Sodium thiosulphate (63g in 250 ml water).
- **b.** 1N Sulphuric acid,
- **c.** 10% Formaldehyde,
- **d.** Rosalic acid

4.Procedure:

1.Take 50 ml of 10% Formaldehyde in a conical flask and add to it neutralized rosalic acid and 25ml of Sodium thiosulphate.
2.Allow it to stand for 10 minutes and then titrate it with 1N Sulphuric acid till color disappears.
3.Note down the volume as sample burette reading.
4. Carry out blank titration and note down the burette reading.

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5.Observation:

Sr.No.	Sample burette reading (ml)	Blank burette reading(ml)
1.		
2.		
3.		
4.	Mean burette reading ---------- ml	Mean burette reading ---------- ml

Sample Burette reading (V1): ml

Blank burette reading(V2) : ml

6.Sample Calculation:

gm of Formaldehyde /100ml of solution = (Blank volume- Sample volume) x Normality of Sulphuric acid x 1.5

7.Result: The percentage purity of formaldehyde is ----------

8.Conclusion: Formaldehyde content in the given sample is ----------.

9. Applications:

10.Assignment Questions:

a.Explain in brief chemical properties of formaldehyde.
b.Explain briefly the various methods of preparation of formaldehyde.

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ACADEMIC YEAR: 2019-20 **PART:I**

COURSE:Organic Chemistry **COURSE COORDINATOR:Ms.A.S.Dutta**

Experiment No.-6

1.Aim: To determine the melting point & boiling point of given sample

2.Apparatus:

a.Thermometer
b.Fusion tube
c.Thermometer
d.Thiele`s tube
e.Capillary tube

3.Chemicals:

- **a.** Sample
- **b.** Paraffin oil

4.Procedure:

1. Fill the paraffin oil in the Thieles' tube approximately 3/4th of Thiele's tube.
2. Take a sufficient amount of sample (solid/liquid) in fusion tube.
3. Tie fusion tube with thermometer bulb by means of thread and suspend it freely in oil containing Thiele's tube containing paraffin oil.
4. Care must be taken that it should not touch to the Thiele's tube and oil should not enter in fusion tube.
5. Heating should be homogeneous at the rate of 2°C/min
6. When the sample (solid/liquid) starts melting/ boiling, note down the temperature as melting/ boiling point .

5.Observation:

The melting point of sample is = ^{o} C

The boiling point of sample is = ^{o} C

6.Result:

The melting point of sample is = ^{o} C

The boiling point of sample is = ^{o} C

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ACADEMIC YEAR: 2019-20 **PART:I**

COURSE:Organic Chemistry **COURSE COORDINATOR:Ms.A.S.Dutta**

Experiment No.-7

1.Aim: To prepare the derivatives of organic compound.

2.Apparatus:

a.Beaker
b.Test tube

3.Chemicals:

a. Bromine water,
b. Hydrochloric acid ,
c. Phenol,
d. Sodium hydroxide,
e. Hydroxyl amine hydrochloride,
f. Formaldehyde

4.Procedure:

1. **PHENOL**

BromoDerivatives :-
1.The phenol (0.3) gm is suspended in dilute Hydrochloric (10ml) and bromine water is added drop wise until no more decolourisation occurs.
2.The bromoderivative which precipitates out is filtered off and recrystallized from alcohol.

2. **OXIMES**

1.Hydroxylamine hydrochloride (0.5gm) is dissolved in water (2ml).

2.10% sodium hydroxide (2ml) is taken and carbonyl compound (0.2-0.3 gm) dissolved in alcohol (1-2ml) is added to it.
3.The mixture is warmed on a steam bath and crystals of oxime are obtained by scratching the sides of the test tube with glass rod.
4.The oximes may be recrystallised from alcohol.

5.Observation:

Thus phenol derivative is obtained.

Thus oximes derivative is obtained.

6.Result:

Phenol and oxime derivatives were prepared.

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ACADEMIC YEAR: 2019-20 **PART:I**

COURSE:Organic Chemistry **COURSE COORDINATOR:Ms.A.S.Dutta**

Experiment No.-8

1.Aim: To determine the elements present in the given sample.

2.Apparatus:

a.Beaker
b.Test tube
c.Conical flask
d.Pipette

3.Chemicals:

a. Sodium sulphate
b. Ammonium sulphate
c. Sample
d. Sodium metal
e. Ferrous sulphate
f. Sulphur
g. Potassium chloride
h. Ferric chloride
i. Sulphuric acid
j. Silver nitrate
k. Nitric acid

4.Procedure:

a. **Test for carbon**

Add sodium sulphate to sample followed by ammonium sulphate. Heat it. Add water and then ferrous sulphate.

5.Observation:
1. If blue coloration occurs the carbon is present.
2.If no blue coloration occurs then carbon is absent.

b.Test for Hydrogen:
Sample is heated with sulphur and fumes are collected on filter paper soaked in lead acetate solution.

5.Observation:
1.If brown spot is observed on paper then hydrogen is present.
2.If no brown spot seen on paper then hydrogen is absent.

c.Test for Oxygen:
Soak filter paper in potassium chloride solution and then dip it in sample solution.

5.Observation:
1. If wine red colouration obtained then oxygen is present.
2. If no wine red colouration obtained then oxygen is absent.

Sodium fusion extract:
Fuse sodium metal in a fusion tube , add sample and heat till red hot.
Plunge it in 10ml of water, boil solution and filter it. Filtrate obtained is the extract.

d. Test for Nitrogen :
Add aqueous Ferrous sulphate to extract. Heat it and then cool it and add sulphuric acid and aqueous ferric chloride.

5.Observation:
1. If blue precipitate obtained then nitrogen is present.
2. If no blue precipitate obtained then nitrogen is absent.

e.Test for Halogens:

Add silver nitrate and nitric acid to extract.

5.Observation:

1. If white precipitate obtained then halogen is present.
2. If no white precipitate obtained then halogen is absent.

6.Result:

Sample	Observations	Inferences

7.Conclusion:

The elements present in given sample are:

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ACADEMIC YEAR: 2019-20 **PART:I**

COURSE:Organic Chemistry **COURSE COORDINATOR:Ms.A.S.Dutta**

Experiment No.-9

1.Aim: To determine the functional group of given organic compound.

2.Apparatus:

a.Beaker
b.Test tube
c.Conical flask
d.Pipette

3.Chemicals:

a. Adipic acid
b. Sodium bicarbonate
c. Bromine water,
d. Hydrochloric acid ,
e. Phenol,
f. Sodium hydroxide,
g. Liquid ammonia
h. Silver nitrate
i. Formaldehyde
j. Methanol
k. Sugar
l. Sulphuric acid
m. 2-napthol
n. Ethanol

4.Procedure:

1.Carboxylic Acid:-

Test with 5% aqueous Sodium bicarbonate

R-COOH + NaHCO3 = R-COO-Na+ + CO2 + H2O

Sodium Hydrogen Carbonate reacts with Carboxylic acid to give the Sodium salt of acid and liberates carbondioxide. If the acid is insoluble in water and the reaction is sluggish. Dissolve the acid in methanol and add carefully to a the saturated Sodium Hydrogen Carbonate solution.

Result:- Vigorous effervescence is obtain therefore acid is result.

2. Phenols :-

Phenols are generally reactive towards electrophilic reagent and are readily brominated by Bromine water.

Eg.

Phenols + 3Br2 = 2,4,6 tri-Bromophenol.

Suspend about 0.05g of the compound in 2ml of dilute hydrochloric acid and bromine water is added till constant colour appears. A white ppt of the bromophenol is formed.This solid bromophenol derivatives can be used for the confirmation of the structure of phenols.

Result:- A white precipitate of bromophenol was obtained. Therefore phenol is present.

3. Aldehydes:-

Prepration of Tollens reagent:- Add 1ml of Silver nitrate to a few drops of sodium hydroxide and then add dilute ammonium hydroxide dropwise until the precipitate just dissolves.

Add 2-3 drops of the aldehyde in Methanol to 2-3ml of tollen's reagent contained in very clean test tube. If no reactions takes place in the cold, warm gently in a water bath.

Result:- The silver mirror on the inside of a clean test tube is obtained. Therefore, aldehyde is present.

4. Sugar (Carbohydrates):-

Molisch's Test is general test for carbohydrates.

Molisch Reagent: Prepare 20% solution of 2-Naphthol in ethanol .

Dissolve 20-30mg of compound (sugar) in 2ml water and add 0.5ml of the molisch`s reagent. Pour 2ml of concentrated H_2SO_4 from a dropper carefully down the side of the tube so that the acid form a layer beneath the aq. solution without mixing it.

Result:- A red colour changes to dark purple at the interface therefore sugar and carbohydrates are present

5.Result:

Thus functional groups of aldehydes,acids,phenols and sugars were detected.

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ACADEMIC YEAR: 2019-20 **PART:I**

COURSE:Organic Chemistry **COURSE COORDINATOR:Ms.A.S.Dutta**

Experiment No.-10

1.Aim: To test the solubility of various organic compound in given solvent.

2.Apparatus:- Beakers and Test tubes.

3.Chemicals:-

a. Samples of organic compounds
b. Sodium hydroxide
c. Sodium bicarbonate

4.Observations:

Solubility Test Tables:-

Sr .no.	Reagent and Test	Class	Group of Compounds
1	Soluble in cold or hot water (If the unknown is soluble do not perform solubility test below)	Neutral, acidic or basic (Test with litmus or universal indicator papers)	Lower members of series neutral. e.g alcohol;acidic. e.g Acid,phenols;basic e.g amines
2	Soluble in dil. Hydrochloric acid	Basic	Most amines
3	Soluble in Sodium hydroxide	Acidic	Most acids, phenols
4	Soluble in Sodium bicarbonate	Strongly acidic	Most carboxylic acids
5	Insoluble in water acid & alkali	Neutral	Hydrocarbons, alkyl or aryl halides esters & ethers, alcohols, aldehydes and Ketones.

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5.Result:-

Sample	Soluble In	Inference
A	Hot water	Neutral
B	Benzene	Basic
C	Cyclo Hexanone	Neutral
D	Hot water	Acidic