IV SEMESTER

PRACTICAL MANUAL FOR SECOND B.Sc. CHEMISTRY

(w. e. f. 2020 – 2021)

PRACTICAL – 4 (ORGANIC QUALITATIVE ANALYSIS)

PREPARED BY

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SYLLABUS

LABORATORYCOURSE -IV 30 Hrs (2 H / W)

PRACTICAL COURSE-IV ORGANIC QUALITATIVE ANALYSIS 50M
(At the end of Semester- IV)

Course outcomes

At the end of the course, the student will be able to

- Use glassware, equipment and chemicals and follow experimental procedures in the laboratory.
- Determine melting and boiling points of organic compounds.
- Understand Application of concepts of different organic reactions studied in theory part of organic chemistry.

Organic Qualitative analysis 50M

Analysis of an organic compound through systematic qualitative procedure for functional group identification including the determination of melting point and boiling point with suitable derivatives.

Alcohols, Phenols, Aldehydes, Ketones, Carboxylic acids, Aromatic primary amines, amides and simple sugars

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SCHEME OF VALUATION

Time: 3 Hours **Max. Marks: 50M**

Record	Marks:5M
Viva-Voce	Marks:5M
Practical	Marks:40M

1.	Colour	1 Mark
2.	Structure	1 Mark
3.	Odour	1 Mark
4.	M.P/B.P	5 Marks
5.	Ignition test	2 Marks
6.	Litmus test	1Mark
7.	Solubility	2 Marks
8.	Dectection of extra element	5 Marks
9.	Test for unsaturation	2 Marks
10.	Identification of functional group	4 Marks
11.	Conformation tests for functional group	6 Marks
12.	Systematic recording of observations (Including negative tests)	8 Marks
13.	Naming of Compound	2 Marks

HOD OF CHEMISTRY

Head of the Dept. of Chemistry.
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SYSTEMATIC ORGANIC PROCEDURE

EXPERIMENT	OBSERVATION	INFERENCE
Preliminary tests		
Physical state	Solid: Powder / Crystalline	
	Liquid:Viscous /Non-viscous	
Colour		
Odour		
Litmus test		
Moist blue litmus is placed in a little of the substance	Blue changes to Re	May be carboxylic acid or Phenol
	Blue changes to light red	Compound easily oxidizes to acids (Aldehydes)
Moist red litmus is placed in a little of the substance	Red changes to blue	May be Amine
	No change	Neutral
Ignition test Place a pinch of the substance at the end of a nickel spatula and heat it in naked flame	Burnt with Sooty flame	Aromatic
	Burnt with non-sooty flame	Aliphatic
	Burnt with sugar smell	May be carbohydrate /tartaric acid
Test for Un saturation		
With $KMnO_4$ solution Dissolve a pinch of the substance in 2 mL of water and add 2 % potassium permanganate solution drop wise.	Not decolourised or less than three drops decolourised.	Saturated compound
	More than three drops were decolourised	Un saturated compound
With Bromine water To a little substance bromine in water is added.	decolourised	Unsaturated Compound
Solubility behavior Place a pinch of the substance in few mL of the following solvents separately and shake well.		
1. water		
2. 5 % NaOH solution		
3. 5% $NaHCO_3$ solution		
4. 5 % HCl solution		
5. Conc. H_2SO_4		
Melting point or Boiling point		

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PREPARATION OF SODIUM FUSION EXTRACT

A pea size dry sodium metal is taken into an ignition tube and the tube is heated to redness till the metal melts. The tube is now cooled and a small quantity of the organic substance is introduced into the tube and fused again. The hot ignition tube is plunged into a mortar containing cold water. The solid matter is grinded and filtered. The filtrate is called Sodium fusion extract.

Test for Nitrogen To a few mL of sodium fusion extract add 1 mL of freshly prepared ferrous sulphate solution. A dirty green precipitate is formed. Heat the contents and cool under tap. Add dilute sulphuric acid.	Prussian blue colour $Fe_4[Fe(CN)_6]_3$solution is formed	Nitrogen is confirmed
IDENTIFICATION TESTS FOR FUNCTIONAL GROUPS		
TEST FOR CARBOXYLIC GROUP		
Test with Sodium Bicarbonate To the organic compound add sodium bi carbonate solution.	Effervescence of colourless and odourless gas (CO_2) is evolved.	May be carboxylic acid
TEST FOR PHENOLIC GROUP		
Test with neutral ferric chloride solution Dissolve a pinch of the substance in water or alcohol, and add a few drops of neutral ferric chloride solution.	Violet or wine–red or green or blue colour solution is formed	May be a phenolic compound
	With aqueous solution	
	White precipitate changing to red or violet	α-naphthol
	Blue	Resorcinol
	With alcoholic solution	
	Green	β-naphthol
	Bluish violet	α-naphthol
TEST FOR CARBOHYDRATE		
Molish test To the organic substance add water and 4 drops of Molish reagent (10% solution of α-naphthol in alcohol or in chloroform). Allow few mL of Conc. $H_2SO_4$to flow down the side of the inclined tube	A red ring appeared at the junction of the liquids	May be carbohydrate

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TEST FOR CARBONYL COMPOUNDS		
Test with 2,4-DNP To the organic substance add few mL of 2,4-dinitrophenyl hydrazine and shake well.	A yellow crystalline precipitate is formed	May be carbonyl compound
Test with Sodium bisulphite To a pinch of the substance Sodium bisulphite solution is added.	White ppt. formed	May be carbonyl compound
TEST FOR AMIDES		
Test with sodium hydroxide Heat a pinch of the organic substance with few mL of NaOH solution.	Ammonia gas is evolved	May be amide
TEST FOR ALCOHOLS		
1. **Action of sodium metal** 2. **Iodoform test** To afew mL of ethanol, iodine water and NaOH solution are added drop by drop and shaken well.	Reacts vigorously giving H_2 Yellow crystals of iodoform separated	May be alcohols Ethanol
TEST FOR PRIMARY AMINES		
Carbyl amine test Heat a pinch of the substance with 2 drops of chloroform and 1 mL of alcoholic KOH	Offensive odour of phenyl isocyanide (carbylamine) is formed	May be primary amine.
CONFIRMATORY TESTS		
TESTS FOR CARBOXYLIC ACIDS		
Phenolphthalein test Dissolve a pinch of the substance in water or alcohol, add one drop of phenolphthalein and then add 5% sodium hydroxide drop wise.	Pink colour solution is formed after the addition of sodium hydroxide	Carboxylic acid is confirmed
Ester formation test To the pinch of the substance add ethyl alcohol and Conc.H_2SO_4 and slowly heat it for few minutes. Transfer the contents into a beaker containing sodium carbonate solution.	Fruity smell is evolved	Carboxylic acid is confirmed

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TESTS FOR PHENOLIC GROUP		
Libermann's test To a pinch of the substance add conc.H_2SO_4 and shake well to dissolve the substance. Add a few crystals of solid sodium nitrite and shake well. Pour the contents in a beaker half-full of water	Red colour is formed. On adding NaOH solution the colour changed to blue green	Phenolic compound
Phthalein test Heat a pinch of the substance two pinches of phthalic anhydride and 2 drops of conc.H_2SO_4 in a dry test tube. Cool the contents and add NaOH solution to make it alkaline and pour a few drops of this solution in water taken in a small beaker.	Red colour	Phenol
	Green	α-naphthol or β-naphthol
	Fluorescent green	Resorcinol
TESTS FOR CARBOHYDRATE		
Test with Fehling's reagentor Benedict's reagent (general test for reducing sugars)Place a pinch of the substance and few mL of water in a test tube. Place the reagent in another test tube and heat it to gentle boiling and add this hot reagent drop wise to the boiling solution of the substance.	The blue colour of the reagent disappeared and a yellow precipitate changing to red, settled down	Reducing carbohydrates
Test with Barfoed's reagent Place a pinch of the substance few mL of water and few mL of the reagent in a test tube and heat it in a beaker of boiling water.	Red precipitate formed within 2 minutes.	Monosaccharide(glucose or fructose)
	Red precipitate formed in about 10 minutes.	Disaccharide(sucrose)
Test with Tollen's reagent To the substance add Tollen's reagent and warm in a beaker of water.	A silver mirror is formed on the inside of the test tube.	Carbohydrate
Osazone test To a little glucose solution, freshly prepared phenyl hydrazine reagent is added and heated on a water bath.	Yellow crystals are formed	Carbohydrate

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TESTS FOR ALDEHYDES and KETONES		
Test with Schiff's reagent Add a pinch of the substance to few mL of the Schiff's reagent and shake the mixture in the cold.	Pink colour	Aldehyde (aliphatic)
	Without effect	Ketone or aromatic aldehyde
Test with Fehling's reagent: Add a pinch of the substance to Fehling's solution and heat it on a boiling water bath for 5 minutes.	Yellow or red precipitate	Aldehyde
	No precipitate	Ketone
Test with Tollen's reagent To the substance add Tollen's reagent and warm in a beaker of water.	A silver mirror is formed on the inside of the test tube.	Aldehyde
	No silver mirror	Ketone
Iodoform test Dissolve the substance in water and add a few drops of potassium iodide-iodine reagent and then add NaOH solution drop wise.	Yellow crystalline precipitate is formed(Iodoform)	May be acetone or acetaldehyde or acetophenone.
TESTS FOR AMIDES		
Test with Nitric acid Dissolve a pinch of the substance in a little of hot water and cool. Add 1 mL of Conc.HNO_3 and stir well by means of glass rod.	Crystalline white precipitate	May be Urea
	No crystalline white precipitate	May be acetamide or benzamide.
Biuret test In a dry test tube a pinch of the substance and heat it just above its melting point and observe the evolution of ammonia the liquid freezes to form biuret. Dissolve the solid biuret in warm NaOHsolution,cool,and add very dilute solution of copper sulphate drop wise	Purple colour is formed	Amide is Urea

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TESTS FOR PRIMARY AMINES		
Diazotisation test(Dye test) Dissolve a pinch of the substance in dilute HCl, cool under tap (preferably in ice) and add a few drops of $NaNO_2$ solution. Pour the contents in a beaker containing alkaline β-naphthol solution	Bright orange red dye is formed	Aromatic primary amine
Nitrous acid test Dissolve a pinch of the substance in few mL of dilute HCl and cool. Add 10% sodium nitrite solution or crystals.	Brisk effervescence (nitrogen) is formed	Aliphatic primary amine

REPORT

The given compound is ……

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