East Point College of Pharmacy

East Point Campus, Jnana Prabha, Virgo
Nagar PostBengaluru – 560049, Karnataka

Approved
by
Pharmacy Council of India, New Delhi

Affiliated
to
Rajiv Gandhi University of Health SciencesKarnataka
Bengaluru – 560041 India

LAB MANUAL

PHARMACEUTICAL ORGANIC CHEMISTRY-I

B. PHARM 2nd SEMESTER

EAST POINT COLLEGE OF PHARMACY
East Point Campus, Jnana Prabha, Virgo Nagar Post,
Bengaluru – 560049, Karnataka

B Pharmacy

Program Outcomes (PO's)

PO 1- Pharmacy Knowledge

Possess knowledge and comprehension of the core and basic knowledge associated with theprofession of pharmacy, including biomedical sciences; pharmaceutical sciences; behavioral, social, and administrative pharmacy sciences; and manufacturing practices.

PO 2- Planning Abilities

Demonstrate effective planning abilities including time management, resource management,delegation skills and organizational skills. Develop and implement plans and organize workto meet deadlines.

PO 3- Problem analysis

Utilize the principles of scientific enquiry, thinking analytically, clearly and critically, whilesolving problems and making decisions during daily practice. Find, analyze, evaluate and apply information systematically and shall make defensible decisions

PO 4- Modern tool usage

Learn, select, and apply appropriate methods and procedures, resources, and modernpharmacy-related computing tools with an understanding of the limitations.

PO 5- Leadership skills

Understand and consider the human reaction to change, motivation issues, leadership and team-building when planning changes required for fulfillment of practice, professional and societal responsibilities. Assume participatory roles as responsible citizens or leadership roles when appropriate to facilitate improvement in health and wellbeing.

PO 6- Professional Identity

Understand, analyse and communicate the value of their professional roles in society (e.g.health care professionals, promoters of health, educators, managers, employers, employees).

PO 7- Pharmaceutical Ethics

Honor personal values and apply ethical principles in professional and social contexts. Demonstrate behaviour that recognizes cultural and personal variability in values,communication and lifestyles. Use ethical frameworks; apply ethical principles while making decisions and take responsibility for the outcomes associated with the decisions

PO 8- Communication

Communicate effectively with the pharmacy community and with society at large, such as,being able to comprehend and write effective reports, make effective presentations and documentation, and give and receive clear instructions

PO 9- The Pharmacist and society

Apply reasoning informed by the contextual knowledge to assess societal, health, safety andlegal issues and the consequent responsibilities relevant to the professional pharmacy practice.

PO 10- Environment and sustainability

Understand the impact of the professional pharmacy solutions in societal and environmentalcontexts, and demonstrate the knowledge of, and need for sustainable development.

PO 11- Life-long learning

Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change. Self-access and use feedback effectively from others to identify learning needs and to satisfy these needs on an ongoing basis.

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Programme Specific Outcomes (PSO's)	
PSO 1	Acquire a thorough foundational knowledge in pharmaceutical sciences, including pharmacology, pharmaceutics, medicinal chemistry, and pharmacognosy, to excel in further academic pursuits
PSO 2	Gain expertise in the application of contemporary pharmaceutical techniques and technologies, enhancing employability across various sectors including the pharmaceutical industry, academia, and research institutions.
PSO 3	Equip with entrepreneurial skills and knowledge of pharmaceutical business management, including market analysis, product development, regulatory affairs, and financial planning, to initiate and run successful ventures in the pharmacy sector

Course Outcomes (CO's)	
Code: BP208P Pharmaceutical Organic Chemistry-I	
CO 1	Identify the organic compounds by systematic qualitative analysis
CO 2	Determine the boiling /melting point of organic compounds and derivatives
CO 3	Preparation of suitable solid derivatives from organic compounds
CO 4	Construction of molecular models

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EAST POINT COLLEGE OF PHARMACY
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Experiment. No 1

PRELIMINARY TEST

AIM: To Perform the Preliminary tests for the given Organic Compound

EXPERIMENT	OBSERVATION	INFERENCE
A. PRELIMINARY TEST		
1. Physical state	Solid Liquid	Maybe Carbohydrates, amides, aromatic carboxylic acids etc Maybe aldehydes, alcohols, aliphatic acids, nitro compounds, esters etc
2. Colour	Coloured compounds. Colourless compounds.	
3.Odour	i.Odour of bitter almonds. ii.Fruity odour. iii.Odour of carbolic soap. iv.Fishy or Unpleasant odour. v.Pleasant odour.	I. It may be benzaldehyde, nitrobenzene. ii.Ester. iii.Phenol. iv. All Amines. v.Alcohols or Halogenated hydrocarbon.
4.Ignition Test Take a little amount of sample on Nickel spatula and burn it on a bunsen burner.	If it burns with sooty flame. If it burns without sooty flame.	Aromatic compound. Aliphatic compound.
5.Test for Unsaturation. [Bayer's Test] Substance +0.5ml of acetone+0.5ml of sodium carbonate solution(Na_2CO_3)+Potassium permanganate Solution($KMnO_4$)drop wise and shake well.	Decolourisation of Potassium permanganate. Retaining colourisation or No colourisation of Potassium permanganate.	Unsaturated compound. Saturated compound.
6.Litmus Test Dissolve or suspend a small amount of sample in water and add a piece of red litmus/blue litmus paper.	Red litmus turns Blue. Blue litmus turns Red. No change in colour.	Sample is alkaline(amines etc). Sample is acidic(Carboxylic acids, Phenols, Alcohols etc). Sample is neutral (aldehydes,ketones,carbohydrates etc).
B.TEST FOR SPECIAL ELEMENTS [LASSAIGNE'S TEST]		

Place a small piece of freshly cut dry sodium in a dry fusion tube. Add a small amount of sample into the fusion tube. Heat the fusion tube to red hot. Once it is red hot transfer the fusion tube content to a mortar containing a small amount of water (1/2 or 3/4 test tube of distilled water). Crush the sodium fusion, and filter the solution. The filtrate is called Sodium Fusion Extract (SFE), which is further used to test Nitrogen, Sulphur & Halogens.		
1. Test for Nitrogen Take 2 ml of stock soln(SFE) in a test tube. Add a few crystals of Ferrous sulphate. Boil gently, cool, and acidify with dil sulphuric acid till the solution is clear. At last add 1ml of neutral ferric chloride solution ($FeCl_3$).	Blue or Green colour. No Blue or Green colour.	Nitrogen Present. Nitrogen Absent.
2. Test for Sulphur a.Take 2ml of stock solution(SFE) into a test tube. Add 2-3 drops of freshly prepared sodium nitroprusside soln+1 drop of dil.NaOH.	Purple colour. No Purple colour.	Sulphur present. Sulphur absent.
b. Take 2ml of SFE in a test tube + 2-3 drops of Lead acetate solution+ 1 drop of acetic acid (CH_3COOH).	Brown/Black colour. No Brown/Black colour.	Sulphur present. Sulphur Absent.
3. Test for Halogens(Cl,Br,I) Take 1ml of SFE+1ml of dil. HNO_3 boil well & cool. Then add 10% $AgNO_3$ Solution.	No Precipitate formed. Precipitate formed. White colour ppt. Soluble in NH_4OH and insoluble in dil.HNO_3. Pale yellow ppt. Sparingly soluble in NH_4OH and insoluble in dil. HNO_3. Yellow ppt. Insoluble in both NH_4OH and dil.HNO_3.	Halogens Absent. Halogens Present. Chlorine Present. Bromine Present. Iodine Present.

C.SOLUBILITY TABLE

I	II	IIIa	IIIb	IV	V	VI	VII
Soluble in both water and ether.	Soluble in water but insoluble in ether.	Soluble in both 5% NaOH & $NaHCO_3$.	Soluble in 5% NaOH but insoluble in $NaHCO_3$.	Soluble in dil. HCl	Compounds not containing N or S		Compounds containing N or S, soluble in Conc. H_2SO_4
					Soluble in Conc.H_2SO_4	Insoluble in Conc.H_2SO_4	
Ex-Resorcinol, Oxalic Acid.	Ex-Urea, Thiourea, Dextrose, Fructose.	Ex-Benzoic acid, Cinnamic acid, Salicylic acid.	Ex-Phenol.	Ex-Aniline.	Ex-Ethanol, Benzaldehyde, Acetophenone, Ethyl acetate.	Ex-Benzene, Chlorobenzene.	Ex-Acetanilide, Nitrobenzene.
Lower members of 1.Acids. 2.Phenols. 3.Aldehydes and ketones. 4.Esters. 5.Alcohs.	1.Amides and Ureas. 2.Carbohydrates	1.Acids and Phenolic acids	1.Phenols	1.Amines (Primary, Secondary, Tertiary).	1.Aldehydes and ketones. 2.Esters. 3.Alcohols.	1.Halogenated hydrocarbons. 2.Aromatic hydrocarbons.	1.Amides. 2.Anilides. 3.Nitro compounds.

Experiment.No:2

QUALITATIVE TEST FOR PHENOLS

AIM: To Perform the Qualitative tests for Phenols

EXPERIMENT	OBSERVATION	INFERENCE
A. To the sample solution in a dry test tube add 1-2 drops of neutral ferric chloride ($FeCl_3$).	It gives a violet colour	Resorcinol is present.
B. Phthalein Test Take the sample in a dry test tube and add phthalic anhydride, few drops of Conc. H_2SO_4.Heat, cool and transfer into a beaker which contains 5ml of water and 5ml of dil.NaOH.	It forms a yellowish-green fluorescence.	Resorcinol is confirmed.
C. Liberman n's Test Dissolve the sample in conc.H_2SO_4, add $NaNO_2$ to the solution. Shake thoroughly and warm the solution. Transfer to a beaker which contain 10ml of water and make the soln alkaline by adding 1-2 drops of dil. NaOH.	Gives a pink colour.	Resorcinol is confirmed.

Experiment.No:3

QUALITATIVE TEST FOR CARBOHYDRATES

AIM: To Perform the Qualitative tests for Carbohydrates

EXPERIMENT	OBSERVATION	INFERENCE
A. Substance + Conc. H_2SO_4 warm.	Charring without evolution of gas.	Carbohydrates are present.
B. Molisch's Test 0.1g substance in 3ml of water + 2-3 drops of Molisch's reagent (10% shake add 2ml of conc. H_2SO_4 along the sides of the test tube without shaking.	Violet ring at the junction and deep violet colour on shaking.	Carbohydrates are present.
C. Fehling's Test Equal volumes of fehling's solution A and B + dilute solution of the substance, boil.	Red precipitate.	Reducing Sugar (Glucose, Fructose, Lactose, Maltose).
D. Barfoed's Test 1ml dilute solution of substance + 1ml fresh barfoed's reagent (0.3g neutral copper acetate in 5ml cold 1% acetic acid) – heat in a water bath.	Yellowish red ppt after prolonged heating.	Disaccharides.
E. Osazone formation Substance + sodium acetate + phenyl hydrazine HCl - heat in a water bath.	Yellow crystalline ppt of osazone.	Carbohydrates.
F. Benedict's reagent Test 1ml of dilute soln of substance + benedict's reagent. Keep in a water bath, cool.	Orange or red ppt.	Carbohydrate is a reducing sugar.
G. Tollen's reagent Test 1ml of dilute soln of substance + tollen's reagent. Keep in a water bath, cool.	Silver mirror or black ppt.	Carbohydrates is a reducing sugar.
H. Seliwanoff's Test 1ml dilute soln of substance + seliwanoff reagent. Keep in a water bath, cool.	No Red colour.	Aldosugar.
I. Polysaccharides Test 1ml dilute soln of substance + Iodine solution.	No Blue colouration.	Polysaccharides absent.

Experiment.No:4

QUALITATIVE TEST FOR AMIDES/ UREA

AIM: To Perform the Qualitative tests for Carbohydrates

EXPERIMENT	OBSERVATION	INFERENCE
A. Boil 0.3g of the substance + 3ml of 10% NaOH smell. Hold a moist red litmus paper or glass rod dipped in Conc.HCl near the mouth of test tube.	Smell of Ammonia. Red litmus turns to blue. Dense white fumes of NH4Cl near the glass rod.	Amide or urea present.
B. Boil a small quantity of the substance with 3ml of 1:1 HCl.	Acetic acid evolved.	Amide or urea present.
C. Hoffmann reaction 1ml of NaOH + bromine water dropwise till yellow colour persists. Add 0.1g of the substance.	Decolourised with evolution of Nitrogen.	Urea is present.
D. Nitrous acid Test Dissolve 0.3g of the substance + 3ml of dil. HCl + 3ml of 10% $NaNO_2$(Sodium Nitrite).	Effervescence with evolution of nitrogen.	Urea is present.
E. Urea Nitrate Test Dissolve 0.3g of the substance + 3ml of water + 1ml Conc.HNO_3. Scratch the sides the test tube if necessary.	Crystals of Urea nitrate separates out.	Urea is present.
F. Urea Oxalate Test Dissolve 0.3g of the substance + 3ml of water + 1ml Conc. Oxalic acid solution. Scratch the sides the test tube if necessary.	Crystals of Urea oxalate separates out.	Urea is present.
G. Biuret Test Boil 0.3g of the substance in dry test tube + Dissolve in 3ml of dil. NaOH. Add solution of a very dil.$CuSO_4$ dropwise.	Purple/Violet colour.	Urea is confirmed.
H. Test for Thiourea a. Boil 0.3g of the substance in 3ml of dil. NaOH. Heat, add solution of lead acetate. b. Boil 0.3g of the substance and NaOH in dry test tube + Dissolve in water. Add solution of aqueous $FeCl_3$ solution.	Brown/Black colour. Blood red colour.	Thiourea present. Thiourea present.

Experiment.No:5

QUALITATIVE TEST FOR CARBOXYLIC ACIDS

AIM: To Perform the Qualitative tests for Carboxylic acids

EXPERIMENT	OBSERVATION	INFERENCE
A. 0.3g of the substance + 3ml of $NaHCO_3$.	Effervescence and clear solution.	May be Carboxylic acid.
B. Esterification Test Substance + 1ml ethanol + 1ml Conc. H_2SO_4- Heat for 5min cool-pour into bicarbonate solution.	Fruity odour of ester.	Carboxylic acid present.
C. Substance + water shake well. Add 2 -3 drops of phenolphthalein and then small amount of dil. NaOH solution.	Pink colour disappears.	Carboxylic acid present.
D. Neutral $FeCl_3$ Test Substance + neutral $FeCl_3$ solution.	Violet colour or blue green colour. Forms a buff colour ppt.	Pheolic acid present. Benzoic Acid present.
E. $KMnO_4$ Test To substance add a few drops of $KMnO_4$ soln. Add dil.H_2SO_4 and heat.	It gives a smell of bitter almond.	Cinnamic Acid present.

Experiment.No:6

QUALITATIVE TEST FOR PHENOLS

AIM: To Perform the Qualitative tests for Phenols

EXPERIMENT	OBSERVATION	INFERENCE
A. Bromination Test Substance in water + Br_2 water drop by drop.	A green or blue colour changes to red on dilution and blue green in alkali.	Phenols present.
B. Libermann's reaction Substance + few crystals of $NaNO_2$ + few drops of Conc.H_2SO_4 warm cool-pour into cold water add NaOH.	White crystalline ppt.	Phenols present.
C. Scotten –baumann Reaction Substance + NaOH + acetyl or Benzoylchloride – Heat and pour into water.	Blue violet coloration.	Phenols present.
D. Neutral $FeCl_3$ Test Substance + $FeCl_3$ solution.	Greenish colour ppt.	Phenols present.
E. Phthalein Test Substance + Phthalic acid(2:1 ratio) + 2 drops of Conc. H_2SO_4 heat gently cool and pour into dil. NaOH solution.	Intense yellow or orange fluorescence.	Phenol present.

Experiment.No:7

QUALITATIVE TEST FOR ANILINE

AIM: To Perform the Qualitative tests for Aniline

EXPERIMENT	OBSERVATION	INFERENCE
A. Acetylation Substance + acetyl chloride dropwise – shake.	Vigorous reaction, solid separates.	1^0 or 2^0 amines present.
B. Carbylamine reaction Substance + 2 drops of $CHCl_3$ + 1ml alcoholic KOH warm.	Unpleasant odour.	1^0 amines present.
C. Diazotisation Substance in dil. HCl – cool to 5^0C- pinch of $NaNO_2$. Pour the above reaction mixture into ice cold beta – Naphthol in NaOH.	Orange Dye.	Aromatic 1^0 amine present.
D. Sample + Conc. HCl + sodium nitrite.	Clear yellow solution Turns starch iodide paper blue.	Aromatic 1^0 amine present.

Experiment.No:8

QUALITATIVE TEST FOR ALDEHYDES

AIM: To Perform the Qualitative tests for Aldehydes

EXPERIMENT	OBSERVATION	INFERENCE
A. 2,4-DNP Test 0.2g substance in 3ml dil. HCl + 2ml solution of 2,4-dinitrophenyl hydrazine in dil. HCl, shake well , allow for 5mins.	Yellow orange or red crystalline ppt. at once or on gentle warming on a water bath.	Aldehyde or Ketone present.
B. Sodium Bisulphite Test Equal amount substance and conc. Aq. solution of sodium bisulphate.	Pale yellow crystalline solid. (**Exception Acetophenone**).	Aldehyde or Ketone present.
C. Schiff's Reagent Test 2ml substance + 2ml schiff's reagent – shake for 2mins.	Immediate pink or red colour.	Aldehydes.
D. Fehling's solution Test Equal volumes of fehling's solution **A & B** + 0.2 g substance – boil.	Blue colour changes to reddish brown ppt.	Benzaldehyde present.
E. Tollen's Reagent Test To 2ml of tollen's reagent (1ml of AgNO3 soln. + 2 drops of NaOH + NH4OH till brown ppt. just dissolves). Add 2-3 drops of substance – keep the test tube in hot water bath.	Shining silver mirror or black ppt.	Benzaldehyde present.

Experiment.No:9

QUALITATIVE TEST FOR KETONES

AIM: To Perform the Qualitative tests for Ketones

EXPERIMENT	OBSERVATION	INFERENCE
A. 2,4-DNP Test 0.2g substance in 3ml dil. HCl + 2ml solution of 2,4-dinitrophenyl hydrazine in dil. HCl, shake well, allow for 5 mins.	Yellow orange or red crystalline ppt. at once or on gentle warming in a water bath.	Aldehyde or Ketone present.
B. Sodium Bisulphite Test Equal amount substance and conc. Aq. solution of sodium bisulphate.	Pale yellow crystalline solid. (**Exception Acetophenone**).	Aldehyde or Ketone present.
C. Schiff's Reagent Test 2ml substance + 2ml schiff's reagent – shake for 2mins.	No Immediate pink or red colour.	Ketone present.
D. Legal's Test Substance + Sodium nitroprusside solution + dil. NaOH. Shake well + glacial acetic acid.	Wine Red colour.	Acetophenone present.
E. Zimmermann Test 1ml of sample + 1ml of alcohol + 0.1g of meta dinitrobenzene + NaOH soln – shake well.	Immediate violet colour.	Acetophenone present.
F. Iodoform Test Substance + Iodine in 20% KI soln. Warm, add more iodine soln till the colour persists + 1ml of 10% NaOH soln.	A yellow ppt. of iodoform.	Acetophenone confirmed.

Experiment.No:10

QUALITATIVE TEST FOR ALCOHOLS

AIM: To Perform the Qualitative tests for Alcohols

EXPERIMENT	OBSERVATION	INFERENCE
A. Reaction with sodium Take 1ml of the compound in a dry test tube, and dissolve in dry benzene. Add a small piece of freshly cut sodium metal.	The effervescence of hydrogen gas evolved.	The alcohol group is present.
B. Reaction with acetic anhydride Take 1ml of compound in a dry test tube. Add equal amount of acetic anhydride and 2 drops of Conc. H_2SO_4. It is warmed and poured into a beaker containing 20ml of $NaHCO_3$ soln.	Fruity Odor is obtained.	Alcohol group is present.

Experiment.No:11

QUALITATIVE TEST FOR ESTERS

AIM: To Perform the Qualitative tests for Esters

EXPERIMENT	OBSERVATION	INFERENCE
A. Hydrolysis Reaction Take 1 ml of compound in a test tube and add 2 drops of NaOH soln and a drop of Phenolphthalein indicator. Heat to this and add 3ml of Conc.HCl, heat and cool.	White ppt. is observed.	Aromatic Ester present.
B. Hydroxamine Acid Test Take 2 drops of the compound in a test tube. Add 3ml of hydroxylamine HCl soln and a drop of Phenolphthalein indicator. To the resulting mixture, add alcoholic KOH till it gets pink colour. Boil & cool. Add 3ml of 2N HCl & 5 drops of $FeCl_3$ soln.	A purple colour appeared.	Esters are present.

Experiment.No:12

QUALITATIVE TEST FOR NITRO COMPOUNDS

AIM: To Perform the Qualitative tests for Nitro compounds

EXPERIMENT	OBSERVATION	INFERENCE
A. Mulliken's and Barker's Test Substance + 2ml ethanol + 1ml $CaCl_2$ solution + pinch of Zn dust or tin boil for 5min cool and filter into 2ml of tollen's reagent. Heat on water bath if necessary.	Black ppt.	Nitrocompond present.
B. Acid Reduction Test Substance + conc. HCl + pinch of Zn dust boil, cool perform dye test. Add 0.1g of $NaNO_2$ soln, cool at 0-5^{0}c and add B-Naphthol soln. in NaOH.	Red colour Dye.	Nitrocompond present.
C. Janowsky Reaction Substance + 5ml acetone + 2ml of 5% NaOH. Shake well.	Faint yellow colour.	Mono Nitrocomponds present.
D. Substance + $FeSO_4$ crystals + dil. $H2SO_4$ + ethanolic KOH, shake well.	Brown ppt.	Mono Nitrocomponds present.

Experiment.No:13

DETERMINATION OF MELTING POINT

AIM: To determine the melting point of the given sample

Apparatus: Thermometer, capillary tube, Burner, Stand, Thread

Chemicals Required:

Liquid paraffin wax, and sample substance

Principle:

Melting point is defined as the temperature at which solid becomes into liquid substances under a pressure of one atmosphere is called melting point.

Melting point is determined one of the most common techniques used to characteristic the organic compound and to check the state of purity. Melting point of a crystalline solid is the temperature at which solid begins to change into liquid state. The purity of the compound has sharp melting point due to which the change from solid to liquid is quick.

Impure sample has lower melting point than that of pure. Its melting range is wide. Both temperature and sharpness of the melting (range) point are the useful criteria of purity.

Procedure:

One end of the capillary tube is sealed by heating, it in the non-luminous portion of the flame as well as continuously rotating heating until it is closed. The open end of the capillary tube is pushed into a small amount of completely dried and finally powdered organic compound which is under examination. The powder is shaking by tapping the sealed end of the capillary tube on the bench. The procedure is repeated until the length of the powder material is 3-4 mm outside of the capillary tube wiped clean.

A thermometer is inserted into a one hold rubber stopper. The capillary tube is tied to the thermometer with a rubber band and a thread. The capillary tube is tied in such a way that it's sealed end & indirect contact with the bulb of thermometer.

The tube is filled with liquid paraffin. The thermometer with the capillary tube is immersed in liquid paraffin in such a way that the open end of the capillary tube and rubber band should be above the level of the liquid paraffin the side arm of the tube is heated at a uniform rate. The flame of the burner & adjusted in such a way that the temperature at which the last crystal disappeared and this melting point is reported.

Report:

The given sample melts at …….

Experiment.No:13

DETERMINATION OF BOILING POINT

AIM: To determine the boiling point of the given sample

Apparatus:

Distillation flask, thermometer, stand, burner

Principle:

Boiling point of the liquid is the temperature at which liquid begins to boil and gets converted into its vapor form. This is usually a characteristic of liquid or solvent in its pure form.

Boiling point involves breaking of oppositely charged ions. This occurs when temperature is reached at which thermal energy of the particle is great enough to overcome cohesive force that hold the molecules. Generally, when reasonable amount of liquid compounds are available boiling point is determined by slowly distilling the material from a sphere shaped flask &regarded the boiling point at the temperature at which the liquids starts distilling for small quantity of liquid. The material should be distilled using boiling point apparatus.

Procedure:

Transfer the given liquid into a distillation flask and add 1 or 2 fragments of porcelain. Arrange the apparatus in such a way that the bulb of the thermometer should be in the centre of the flask & slightly below the side tube heat the flask from a flame and adjust the flame in such a way that the distillate is collected at the rate of 1 or 2 drops / Sec. The temperature will rise rapidly until it is near the boiling point of the liquid then slowly and finally it remains constant.

Record the temperature when in remains the constant. Collect the liquid and continue distillation until only a small volume of liquid remains in the flask. Observe the boiling point.

Report:

The given liquid boils at …

Experiment.No:14

THE PREPARATION OF DERIVATIVES OF ORGANIC COMPOUNDS.

AIM: To prepare derivatives of some class organic compounds.

The preliminary examination and group classification tests indicate the particular class (functional group) to which an unknown organic compound may belong. Further characterization and identification depends on the selection and preparation of a suitable solid derivative and accurate determination of its melting point (best, between 90 -150). The following table lists some of the class of organic compounds and selection of derivatives that may be prepared to characterize them.

Class of Compound	Derivatives
1. Alcohols	3,5-Dinitrobenzoate
2. Phenols	Benzoate, acetate, bromo derivative
3. Aldehydes and Ketones	Semicarbazone, 2,4-Dinitrophenyl-hydrazone, oxime
4. Acids	Anilide, amide, p-toluidine
5. Amines	Benzoyl, Acetyl and Sulponamide derivatives

Methods for the preparation of derivatives.

1. Alcohols:

(i)3,5-Dinitrobenzoates

3,5-Dinitrobenzoyl chloride is mixed with the alcohol (0.5-1ml) in a loosely corked dry test tube and heated on the steam bath for about 10min. Secondary and tertiary alcohol require upto 30mins. On cooling add 10ml sodium hydrogen carbonate solution, stir until the ester crystallises out, and filter at the pump. Wash with a little carbonate solution, water and suck dry. Recrystallise from the minimum hot ethanol or light petroleum. Cool slow to avoid the formation of oily droplets of your ester.

2. Phenols:

(i)Benzoates (Schotten-Baumann method).

To the phenol (0.5g) is added 5% NaOH 10ml in a well-corked boiling tube or a small conical flask. Benzoyl chloride (2ml) is added in small quantities at a time, and the mixture shaken vigorously with occasional cooling under the tap or in ice water.

After 15min the solid benzoate is separates out: the solution should be alkaline at the end of the reaction; if not alkaline, or if the product is oily, add a solid pellet of NaOH & shake again. Collect the benzoate, wash thoroughly with cold water and recrystallize from alcohol or light petroleum.

(ii)Acetates

Acetates of many simple phenols are liquids; however, this is a suitable derivatives for polyhydric and substituted phenols. The phenol (0.5g) is dissolved in 10% NaOH soln. and an equal quantity of crushed ice is added, followed by acetic anhydride (2ml). The mixture is vigorously shaken in a stoppered test tube until the acetic separates. The product is filtered and recrystallized from alcohol.

(iii)Bromo derivative

The phenol (0.3g) is suspended in dil. HCl (10ml) and bromine water added dropwise until no more decolourisation occurs. The bromo derivative which ppt. out is filtered out and recrystallize from alcohols.

3. Aldehydes and Ketones

(i) Semi carbazones

Dissolve semic arbazide HCl (1g) and sodium acetate (1.5g) in water (8-10ml), add the aldehyde or ketone (0.3ml) and shake the mixture for few minutes and then cool in ice water. Filter off the crystals, wash with a little cold water and recrystallize from methanol or ethanol.

(ii) 2,4-dinitrophenyl hydrazones

Suspend 0.25g of 2,4-dinitrophenyl hydrazine in 5ml of methanol and add 0.5ml of conc.H_2SO_4 cautiously. Filter the warm soln. and add the soln. of 0.2g of the carbonyl compound in 1ml of methanol. Recrystallize the derivative from methanol, ethanol or ethyl acetate.

(iii) Oximes

Hydroxyl amine HCl (0.5g) is dissolved in water (2ml). 10%NaOH (2ml) and carbonyl compound (0.2-0.3g) dissolved in alcohol (1-2ml) are added, the mixture warmed on steam bath for 10min and then cooled in ice. Crystallization induced by scratching the sides of the test tube with a glass rod. The oximes may be crystallised from alcohol.

4. Acids:

(i) Amides, Anilides and p-toluidine's

The acid (0.5g) is reflexed with thionyl chloride (2-3ml) in a fume cup board for about 30mins*. It is advisable to place a plug of cotton wool in the top of the reflux condenser to exclude moisture. The condenser is removed and the excess of thionyl chloride is distilled off (b.p.78). The acid chloride thus produced is treated with concentrated ammonia solution(5ml) or aniline (0.5-1ml) or p-toluidine (0.5-1g), when the solid derivative separates out. It is collected and recrystallized from alcohol adding decolourising charcoal if found necessary.

*Alternately used PCl_5 to form the acid chloride.

5. Amines:

(i)Acetyl derivatives(acetamides)

Reflux gently in small dry flask under a dry condenser the amine(1g) with acetic anhydride(3ml) for 15mins. Cool the reaction mixture and pour into 20ml cold water. Boil to decompose the excess acetic anhydride. Cool and filter by suction the insoluble derivative. Recrystallize from ethanol.

(ii)Benzoyld erivaties(benzamides)

Suspend 1g of the amine in 20ml of 5% aqueous sodium hydroxide in well corked flask, and add 2ml of benzoyl chloride (fume hood), about 0.5ml at a time, with constant shaking. Shake vigorously for 5-10mins until the odour of the benzoyl chloride has disappeared. Ensure that the mixture remains alkaline. Filter off the solid derivates, wash with a little cold water and recrystallize from ethanol

(iii)Benzene sulphonamides

To 1g of the amines in 20ml of 5% sodium hydroxide solution in a well corked flask and 1ml of benzene sulphonyl chloride (fume hood). Shake the mixture until the odour of the sulphonyl chloride disappears. Check that the solution is alkaline. Acidify is necessary to obtain the precipitated derivative. Concentrated hydrochloric acid added drop wise should be used. Filter the product, wash with a little cold water and suck dry. Recrystallized from ethanol.

Vision and Mission of the Institution

Vision

The East Point College of Pharmacy aspires to be a globally acclaimed institution, **recognized** for **excellence in** pharmaceutical education, research and nurturing students for **holistic development.**

Mission

M1 Create pharmacy graduates through **quality education**

M2 Promote innovation, **creativity**, and excellence **in teaching**, learning, and **research**

M3 **Inspire** integrity, teamwork, critical thinking**, personal** development, and ethics in **students** and lay **the** foundation for lifelong learning

M4 **Serve** the **healthcare, technological, scientific**, and **economic** needs of then **society**.